MELLON INSTITUTE

Final Report

on

EVOLUTION OF ULTRA-HIGH STRENGTH STEELS, AND RESEARCH ON MATERIALS AND VARIOUS NOVEL TECHNIQUES OF FABRICATION OF HIGH PERFORMANCE ROCKET MOTOR CASES

Mellon Institute Project No. 381-3

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FOREWORD

This report presents information relative to research and development work conducted at Mellon Institute on the evolution of ultrahigh-strength steels, and on materials and various novel techniques of fabrication of high performance rocket motor cases. This task was performed over a three-and-one-half years' period under Navy Bureau of Weapons Prime Contract NOrd-18169. Liaison and technical monitorship during the tenure of this contract were provided successively by Messrs. J. L. Browning, E. Roberts, R. Sanderson, J. Pitsenberger, and H. Bernstein, Special Projects Office, Bureau of Naval Weapons.

The contract was administered through Mr. J. F. Kennedy, Office of Naval Research, University of Pittsburgh, Pittsburgh 13, Pennsylvania, and through the Inspector of Naval Material, Old Post Office Building, Pittsburgh 19, Pennsylvania.

SUMMARY

Technical information and experimental data are provided for a newly developed low-alloy ultrahigh-strength steel Rocoloy 270. This steel is capable of achieving yield strength around 270 Ksi and ultimate tensile strength minimum of 310 Ksi in conjunction with other necessary engineering properties.

Data are obtained of the notch tensile strength, biaxial strength, impact strength, elevated temperature strength, and stress-rupture characteristics and also fatigue properties to demonstrate the suitability of Rocoloy 270 for the manufacture of high performance rocket motor cases and as structural material in designs specifying light-weight, high-strength components. Fabrication characteristics are discussed and compared with other similar steels which include observations on various forming and welding aspects of Rocoloy 270.

Information has been presented on experiments conducted to demonstrate weld grain refinement possibilities with the aid of continuously applied ultrasonic energy to the solidifying weld bead.

Included herein is also a report of work done on producing long continuous fine (0.005 in. dia. or finer) filaments of aluminum alloys and beryllium and on the possibilities of constructing metallic filament wound epoxy bonded scale model missile motor configurations.

ACKNOWLEDGMENTS

The Project Leader wishes to thank all the service department personnel of Mellon Institute who contributed their time and effort to seccessful completion of work on this program. The technical assistance provided by Mr. D. V. Lindh, Project Engineer, deserves special mention. Thanks are due to Mrs. E. P. Hartner for conducting light and electron metallographic work on various experimental steels. Services provided by other Project personnel, particularly by Messrs. E. Vandale, E. Jabo, F. Gasper, and P. Welsh are gratefully acknowledged.

Project personnel acknowledge with gratitude and pride the stimulating support and guidance provided by Dr. H. L. Anthony III, a Director of Research at Mellon Institute, whose technical advice contributed in large measure to success of various tasks in this program.

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INTRODUCTION

The objectives of this program at the time of its inception were to develop (a) a material and (b) fabrication techniques for the construction of Model B POLARIS motor case having a minimum yield strength of 230,000 psi. The original proposal was based on the premise that new steels developed up to 1958 and fabricating techniques employed were unsatisfactory for complicated designs specifying yield strength levels above 200,000 psi and that further intensive research on materials and evaluation of fabricating techniques must be accomplished prior to building full size POLARIS motor cases which are to meet higher performance requirements of the then current and the future designs.

In support of the foregoing, Mellon Institute submitted and secured approval with funds for conducting the following research and development program divided into ten closely integrated phases and carried over a three and one-half years of engineering effort.

Phase I

Conduct studies directed toward the formulation, development and evaluation of ultrahigh-strength steels of the AISI 4100, 4300 and modified low-alloy hot-work die steel types having uniaxial yield strengths at 0.2 per cent offset in the range 210,000 psi to 300,000 psi. Goals to be achieved successively were steels having yield strength capabilities of 230,000 psi, 250,000 psi, 270,000 psi and 300,000 psi.

Phase II

Develop higher biaxial strength, improve notch-toughness and corrosion resistance of steels by using modern vacuum melting techniques, such as, vacuum arc remelting of vacuum induction melted material, double vacuum arc remelting of air arc melted steel, heat treatment of high-strength steels in a vacuum, and warm working meta-stable austenite of martensitic or pseudo-austenitic steels.

Phase III

Conduct fracture toughness studies on steels considered for application in POLARIS motor cases using center and edge notched tensile test specimens of parent materials and welds. Also, evaluate using the 10 inch and 12 1/4 inch diameter ellipsoidal and hemispherical cups, the multiaxial stress withstanding capabilities of above steels and correlate such results with heat treatment (strength-level), notch-strength and fracture toughness, microstructures, decarburization, inclusion content and their distribution, and gas content in the material.

Phase IV

Establish forming and joining techniques for ultrahigh-strength steels developed in this program to include, but not to be limited to deep drawing, shear-forming, welding, forging and roll forming.

Phase V

Study the effect of ultrasonic vibration on grain size and solidification of weldments and investigate the feasibility of using this technique in girth welds made in joining rocket motor case components.

Phase VI

Conduct studies directed toward formulation and development of suitable weld filler materials for useful alloy steels developed in the program using the levitation furnace melted and cast pin test for screening weld wire compositions which are incompatible with materials to be welded.

Phase VII

Construct and hydrostatically test scale model rocket motor chambers of suitable materials in order to determine if the biaxial strength levels could be achieved and that the screening tests described in Phase III could be correlated with the yield and burst strength of full scale chambers.

Phase VIII

Conduct studies to determine the stress-corrosion susceptibility of ultrahigh-strength steels considered for use in the POLARIS program and suggest methods of preventing and/or eliminating factors responsible for causing stress-corrosion.

Phase IX

Review research and development proposals involving materials and fabrication techniques used or suggested for future use for the manufacture of POLARIS motor cases and provide technical assistance and advisory services to the Special Projects Office or their contractors and subcontractors.

Phase X

Prepare monthly progress reports, and other technical reports through literature surveys and attending technical meetings and conferences recommending materials, manufacturing processes and fabricating methods, and such other topics pertinent to and helpful for the development and production of POLARIS and other high-performance missile motor cases and other components.

ACCOMPLISHMENTS OF THIS PROGRAM

Research conducted on above phases has resulted in (1) the development of several ultrahigh-strength steels, (2) important metallurgical achievements and (3) contributions to the technology of constructing high-performance rocket motor cases. Outstanding achievements to note are the following:

1. Ultrahigh-strength low-alloy steels capable of achieving minimum uniaxial yield strength (0.2 per cent offset) levels of (a) 230,000 psi (MX-2, 4137 Co) (b) 250,000 psi (Rocoloy*250, MX-27) (c) 270,000 (Rocoloy 270).

2. Demonstrated through successfully testing several 54 in. diameter rolled and welded POLARIS second stage chambers that biaxial yield strength exceeding 230,000 psi can be reliably secured using sandwich rolled air melted MX-2 sheets and vacuum consumable arc remelted MX-2 sheets and ring forgings.

3. Showed through proof and firing testing of thin wall (0.035 in. side wall) Falcon (M-58) cases that full material capabilities of MX-2 and Rocoloy 250 can be achieved by a combination of fabrication techniques involving deep drawing, ironing and machining of sidewall.

4. Accomplished through technical assistance provided by the Project scientists to ACF Industries, Inc., Milton, Pa., the deep drawing of 54 inch diameter POLARIS second stage chamber halves having integral ellipsoidal heads formed by a reverse drawing operation.

5. Developed a material specification for air melted and consumable arc remelted MX-2, weld filler wire for Aerojet-General Corporation.

6. Demonstrated that significant weld grain and microstructural improvement can be affected by the use of double consumable arc remelted weld filler wire.

7. Effected weld grain refinement with the aid of ultrasonic vibration applied continuously during welding missile motor case components.

8. Completed a program of drawing aluminum alloy 6061-T913, 2024-T4 and 7075-T6 wires 0.005 in. diameter in continuous spooled length from 5000 ft. to 14,000 ft.

9. Produced 0.005 in. diameter wire of commercially pure beryllium in lengths up to 2000 ft.

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*A registered trade name of ultrahigh-strength steels developed at Mellon Institute.

10. Constructed a scale model aluminum filament (0.005 in. dia. wire) wound resin bonded sphere in order to determine the feasibility of winding spheres and other configurations using metallic filaments and epoxy bonding techniques.

SPECIAL REPORTS AND TECHNICAL PAPERS

A technical brochure entitled "MX-2 Ultrahigh-Strength Steel for High Performance Solid Propellant Missile Motor Case Application" was prepared and widely distributed as requested by the Special Projects Office of the Bureau of Naval Weapons. This report summarizes progress made through the first 18 months of the contract period. Repeat summarization of the project effort up to this point will not therefore be attempted.

Published technical papers worthy of mention are the following:

(a) "A New Ultrahigh-Strength Steel for High-Performance Rocket Motor Cases" by G. K. Bhat, American Society for Metals Symposium on High-Strength Steels for the Missile Industry, 1961.

(b) "Evaluation of Biaxial Properties of Rocket Motor Case Materials Using Sub-Scale Specimens" by G. K. Bhat and D. V. Lindh, Seventh Sagamore Ordnance Material Symposium Proceedings, 1960.

SUMMARY REPORT ON PROGRESS MADE DURING THE LAST 24 MONTHS OF THE PROGRAM

A list of alloy steel heats evaluated in this program during the past 24 months is given in Appendix A. Of these, Rocoloy 250 and Rocoloy 270 compositions are of importance. All other heats were rejected after preliminary screening tests. Rocoloy 250 is a slightly modified version of MX-2. In this steel, the carbon has been raised to 0.44 per cent and all other alloy ingredients except sulfur and phosphorus of MX-2 raised by about 10 per cent. However, Rocoloy 270 development almost coincided with that of Rocoloy 250 and, because of the higher strength potential of the former, all later research efforts were expended on full evaluation of various Rocoloy 270 compositions.

ROCOLOY 270

Rocoloy 270[1] is a low-alloy ultrahigh-strength steel capable of achieving in the heat treated condition, yield strength[2] levels in the range 265,000 psi to 275,000 psi, and a minimum ultimate tensile strength exceeding 310,000 psi. Rocoloy 270 is a deep hardening steel. When air cooled from the austenitization temperature, sections up to 5 inches in thickness have been through hardened to Rockwell C 58 minimum. Yet, the carbon content of this steel seldom exceeds 0.45 per cent. A notable feature of Rocoloy 270 is that the chemical composition has been so formulated as to prevent occurrence of embrittlement in the entire tempering range from 400 F through 1200 F. The maximum strength, however, is achieved by tempering Rocoloy 270 at 600 F subsequent to hardening by cooling in air, or by quenching in molten salt or by quenching in oil from the austenitization temperature.

Rocoloy 270 has been designed primarily for application in solid propellant rocket motor casing, aircraft structural components, high pressure gas cylinders, high strength bolts and similar items requiring high-strength, light-weight construction.

This steel offers material and fabrication flexibilities not easily obtainable in any other similar low-alloy steel compositions. The lack of susceptibility of Rocoloy 270 to the several embrittling phenomena found in low-alloy constructional steels, increases its versatility and makes this composition attractive for application at wide yield strength levels ranging

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[1] A registered trademark obtained by Mellon Institute. Patent pending.

[2] Yield strength referred to in the text of this manuscript is computed at 0.2 per cent offset from the load strain curve unless specified otherwise.

from 190,000 psi through 270,000 psi. Thus it is evident that Rocoloy 270 can be successfully employed to cut down inventory of several grades of alloy steels falling in the above yield strength range.

Nominal Chemical Composition Range (in Per Cent) of Rocoloy 270

Carbon	0.39 to 0.45
Manganese	0.40 to 0.80
Silicon	0.90 to 1.30
Chromium	1.15 to 1.60
Nickel	0.75 to 1.10
Molybdenum	0.40 to 0.60
Tungsten	0.25 to 0.40
Vanadium	0.10 to 0.20
Cobalt	1.20 to 1.50

Phosphorus and Sulfur each 0.013 per cent maximum.

Aluminum for grain size control should be added such that the acid soluble aluminum content of the steel could be 0.03 minimum up to a maximum of 0.08.

Manufacture of Rocoloy 270

For high strength structural applications, Rocoloy 270 must be made in the electric arc furnace with expert care so as to produce a clean and unsegregated ingot. If the material is to be heat treated to yield strength levels above 210,000 psi, vacuum consumable arc remelting of the air melted electrodes is strongly recommended. For producing extra-fine quality material, a double vacuum consumable arc remelting procedure is considered most suitable. Other vacuum melting procedures, such as in a vacuum induction furnace and any variation of this basic procedure, has not resulted in a product even equal in quality to that manufactured by double vacuum consumable arc remelting. Data supporting the above are given elsewhere in this report.

Steel companies having most experience in melting and processing Rocoloy 270 are Latrobe Steel Company, Latrobe, Pennsylvania, Universal-Cyclops Steel Corporation, Bridgeville, Pennsylvania, and Vanadium Alloys Steel Corporation, Latrobe, Pennsylvania. There are, however, no limitations as to the capability of other steel manufacturers making and processing Rocoloy 270 into various billet, sheet, and bar products provided the equipment necessary is available.

A good source for Rocoloy 270 weld filler wire is Armetco, Inc., Wooster, Ohio. Experimental coated welding rods have been made by McKay Company, and this company would be a source for Rocoloy 270 coated weld rods.

Forging

Rocoloy 270 ingots for forging must be heated slowly and uniformly to 1950 to 2050 F and worked evenly until temperature drops to around 1700 F. It is undesirable to hot work this alloy below 1700 F. Cooling the forging to room temperature must be done slowly and evenly in a furnace until a temperature around 900 F is reached, whereupon the forging could be cooled faster either by opening the doors of the furnace or by pulling it out of the furnace. For good machinability, including such operations as drilling, sawing, chipping or turning, the forgings must be tempered at 1350 F for 2 to 4 hours depending upon the section thickness.

Annealing

The most desirable normal condition for Rocoloy 270 prior to fabrication, using forming operations, such as, deep drawing, bending, peening, welding, is the fully spheroidized annealed state. The hardness in this condition must not exceed Rockwell B 98. A typical spheroidize annealing cycle is to heat Rocoloy 270 sheet or bar to 1550 F $\pm$ 20 F, hold for two hours, fast cool to 1300 $\pm$ 20 F and hold for 12 to 24 hours followed by moderately slow cool to room temperature.

When forming or deep drawing is performed at room temperature using multiple operations, intermediate recrystallization annealing must be performed by heating the stressed part around 1325 F for at least 15 minutes followed by air cooling to room temperature. Rocoloy 270 does not scale due to this fast heat treatment, therefore, a protective atmosphere furnace is not normally needed.

FUNCTION OF VARIOUS ALLOYING ELEMENTS IN ROCOLOY 270

Rocoloy 270 has an unusual chemical composition because of the addition of tungsten and cobalt to a low alloy steel. A few comments regarding the function of each alloying ingredient, in Rocoloy 270 therefore, appear pertinent.

Carbon

Carbon is necessary in steels to be quenched to full martensitic hardness and also for the formation of various alloy carbides which provide additional matrix strength. The amount of carbon required in this steel was fixed on the basis of carbon necessary to form martensite (which is around 0.25 per cent) and that required for stoichiometric combination with the average individual carbide forming alloying elements present in the steel. Thus approximately 0.42 per cent appears to be an optimum carbon content of Rocoloy 270. At this level, carbon appears not detrimental to the fracture toughness and weldability of this steel.

Manganese

Manganese acts as a potent ferrite strengthener but in ammounts exceeding 0.80 per cent in Rocoloy 270, it tends to decrease ductility and also impairs weldability. Manganese above 0.50 per cent reduces slightly the fracture toughness of the base metal, but more drastically that of the weldments. However, the addition of manganese proportionately increases the endurance limit and improves the corrosion resistance of air melted Rocoloy 270 under certain environments.

Silicon

Silicon is added primarily for its ferrite strengthening effect and also its positive contribution to hardenability. Silicon also acts as retardant of the tempering reaction by delaying the precipitation of cementite. This action permits the steel to be tempered in the range 525 to 625 F, which is the normal embrittling zone for steels such as AISI 4340. Silicon in excess of about 1.50 per cent tends to increase the inclusion content and affects the arc characteristics, especially, uniformity in welding.

Chromium

Chromium in amounts used in Rocoloy 270 confers air hardenable capabilities. Chromium, like silicon, also retards tempering and thus helps maintain strength at high levels. Chromium in alloy constructional steels forms solid solution with both the ferrite and austenite phase of iron. It is a strong carbide former, similar in this respect to molybdenum, tungsten and vanadium. Chromium in combination with carbon and iron forms a series of complex carbides. Stable chromium carbides in steels of the type Rocoloy 270 appear to be Cr_7C_3 and predominently $Cr_{23}C_6$. The latter carbide has some solubility for other alloy carbides. Chromium raises the Ac_3 critical temperature. It also promotes greater depth of hardening in medium carbon low-alloy steels due to its retarding effect on the critical cooling rate. When chromium is present in a steel in combination with nickel, it produces superior mechanical properties. In combination with molybdenum, chromium improves elevated temperature strength in Rocoloy 270.

Nickel

Nickel is added primarily for its contributions to enhancing hardenability, improving toughness and resistance to impact stresses at sub-zero temperatures. In constructional alloy steels it acts as a ferrite strengthner. Nickel, in amounts exceeding 0.70 per cent tends to create certain difficulties in recrystallization of microstructure subsequent to cold deformation. Because nickel markedly increases the elastic limit of a steel, it affects cold deep drawability.

Molybdenum and Tungsten

Molybdenum and tungsten as alloying elements in Rocoloy 270, can form a solid solution with the alloyed ferrite and also form a complex carbide. These alloying elements behave similarly in steels of the type Rocoloy 270. However, the purpose of adding tungsten along with molybdenum is to prevent occurrence of temper embrittlement during tempering in the range 850 to 975 F. Although molybdenum is normally added to chromium-nickel steels to reduce their susceptibility to temper-embrittlement, its effect diminishes and actually reverses itself beyond about 0.5 per cent. Molybdenum also increases the resistance of a steel to tempering and thus helps maintain strength of steels at high levels when tempering is done at 800 F to 1000 F. However, more than 0.5 per cent molybdenum is necessary for the latter purpose. The addition of tungsten to Rocoloy 270 provides strength, renders the steel insensitive to temper embrittlement, and also very markedly improves weldability. Since the amount of tungsten addition is kept low, formation of massive type tungsten carbides is avoided and therefore the hardening temperature need not be raised above 1725 F. The small amount of tungsten added to Rocoloy 270 also raises the grain coarsening temperature, thus permitting higher forging and rolling temperatures. Molybdenum to a large extent and tungsten to lesser extent, intensify the individual influence of other major alloying elements in steel.

Vanadium

Vanadium dissolves to some degree in ferrite, thus imparting strength and toughness to low-alloy steels. Its primary function however is to produce a fine grain structure, especially in the welds, and also prevent grain growth when the steel is heated to high temperature in the austenite region.

Vanadium is a strong carbide former. It raises the critical temperature, contributes to resistance to softening of the steel, and also increases hardenability provided the vanadium enters into solid solution during austenitization. Like molybdenum, vanadium intensifies the individual effects of other major alloying elements.

Cobalt

The influence and function cobalt in low-alloy high strength steels appears to have been least understood. There exists a definite paucity of data regarding the effect of cobalt in steels other than high-speed steels and high temperature alloys.

Cobalt dissolves in the iron matrix and brings about increases in density, strength and hardness. It many ways cobalt behaves similarly to nickel in alloy constructional steels. However, cobalt unlike nickel does not increase the strain-hardening characteristics of the steel during cold deformation. Cobalt also acts as an intensifier of the individual effects of other major alloying elements present in the steel. Cobalt tends to increase solubility of carbon in iron and to promote matrix homogeneity and compliments and slightly extends the effect of silicon in retarding tempering in the range 500 F through 625 F. At higher tempering temperature, cobalt also prevents fast decomposition of chromium and other alloy carbides. Cobalt in Rocoloy 270, unquestionably improves weldability, weld strength and ductility.

Sulfur and Phosphorus

Sulfur and phosphorus in Rocoloy 270 should be each maintained under 0.015 per cent. At these levels their influence on any of the steel characteristics is negligible.

PHYSICAL PROPERTIES

The following physical properties of Rocoloy 270 were determined using samples in the annealed condition.

Specific gravity - 7.71 $\pm$ 0.02 gms/cm^3

Density - 0.279 $\pm$ 0.006 lbs./in.^{3}

Modulus of Elasticity - 29.5 x 10^6 ps

Poisson's ratio range - .276 - .278

Mean coefficient of thermal expansion between 75 F and 600 F - 5.73 x 10^{-6} in/in/F

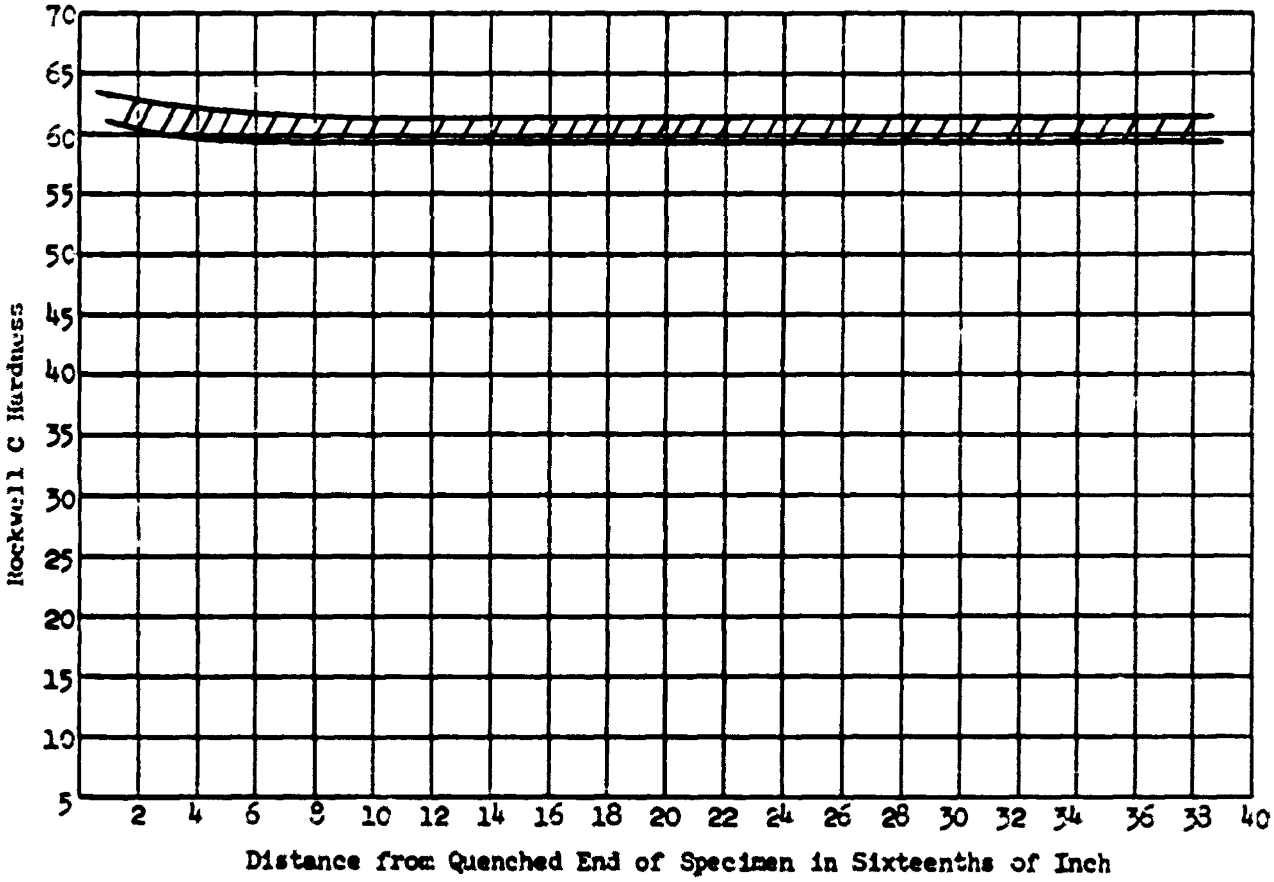
Thermal conductivity - .25 watts/cm$^{\circ}$K

Hardenability

Hardenability data have been obtained for several heats of Rocoloy 270. A typical Jominy hardenability band for Rocoloy 270 is given in Figure 1.

HEAT TREATMENT

Determination of optimum heat treatment for a low-alloy ultra-high-strength steel requires knowledge regarding the critical temperatures, M_s and M_f temperatures, austenitizing and tempering behavior, isothermal



Quenched from 1730 ± 10 F.
ASTM Grain Size 7 to 9.

Figure 1

Hardenability Band for Rocoloy 270

transformation characteristics and the resulting microstructures. Such information has been completely obtained for Rocoloy 270.

Critical and M_s and M_f Temperatures

The Ac_1 and Ac_3 temperatures were determined using the hardness and microscopic technique. Small specimens cut from the annealed sheet or bar product, were heated at 20 F intervals in the range 1400 F through 1680 F, then quenched in water. The hardness of these specimens was measured and the microstructure examined for evidence of freshly formed martensite. The Ac_1 temperature was read at the first evidence of martensite and Ac_3 at around 95 per cent martensite.

The Ac critical temperature ranges for several heats of Rocoloy 270 are:

Ac_1 - 1415 to 1430 F

Ac_3 - 1535 to 1560 F

The M_s and M_f temperatures were determined in a manner similar to the Ac critical temperature, except that only the metallographic technique was used to estimate the degree to which martensite transformation has completed. For Rocoloy 270 and M_s and M_f temperatures are around 560 F and 500 F respectively.

Isothermal Transformation Curve

The isothermal transformation curves for Rocoloy 270, presented in Figure 2, summarizes the reactions which may occur when the steel is cooled from the austenite range to temperatures below A_1 and allowed to transform isothermally. This diagram is helpful in establishing temperatures, time and cooling rates for developing annealing, ausforming, austempering and martempering treatment schedules.

It is to be especially noted that the pearlite reaction completes

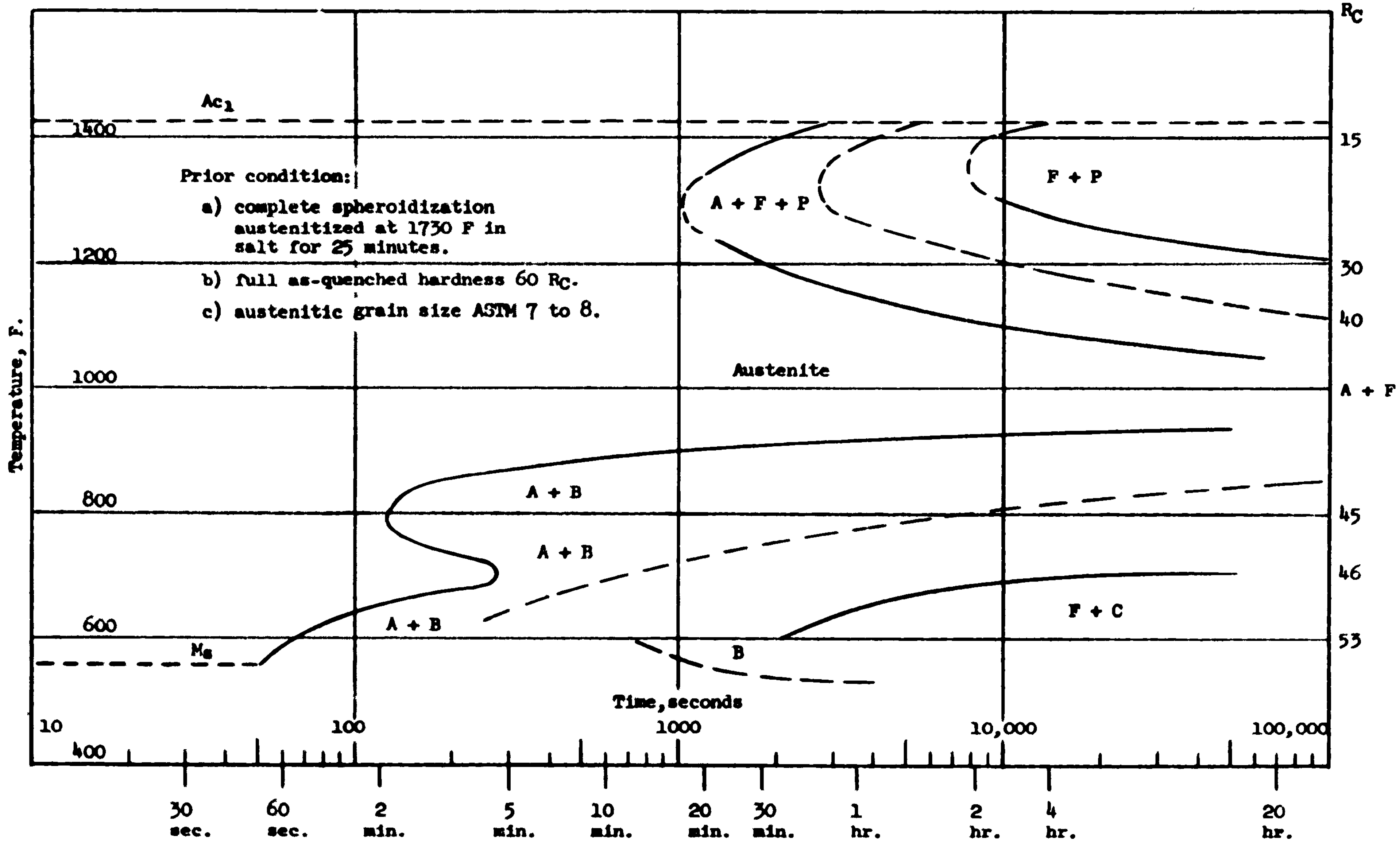


Figure 2 - Time-Temperature-Transformation Diagram for Rocoloy 270.

in about 2 hours and this accounts for the ease of spheroidization of Rocoloy 270. This is believed to be a major asset contributed by cobalt addition to the steel.

Effect of Various Austenitizing and Tempering Temperatures Upon Hardness of Rocoloy 270

The effect of austenitizing Rocoloy 270 at various temperatures upon the resultant as quenched hardness and the retention of stabilized austenite has been studied. Also cumulative tempering in the range from 400 F through 1000 F, using the same specimens has been performed in an effort to develop tempering curves. These curves are plotted in Figure 3. The amount of austenite retained has been estimated by the well known metallographic technique first used by Greninger and Triono[1].

The optimum austenitization temperature and time, determined for several heats of Rocoloy 270 is 1725 ± 10 F and 35 minutes per inch of section thickness up to 4 inches. Retained austenite as a result of this austenitization procedure is less than 1 per cent.

Double tempering for two consecutive 1-1/2 hour periods with intermediate air cooling to room temperature is recommended. If section thickness is over 2 inches triple tempering for three consecutive 1-1/2 hour periods is considered desirable.

Microstructures

Microstructures of Rocoloy 270 in the annealed, hardened and tempered conditions have been studied using both light and electron microscopes. These are shown in Figures 4 through 8. It is a general observation of these studies that the tempered microstructures are extremely fine as to grain size and carbide size. This offers a partial explanation for the high strength, good ductility, toughness and uniformity of properties from heat to heat.

The sequence of microstructional changes occurring during tempering

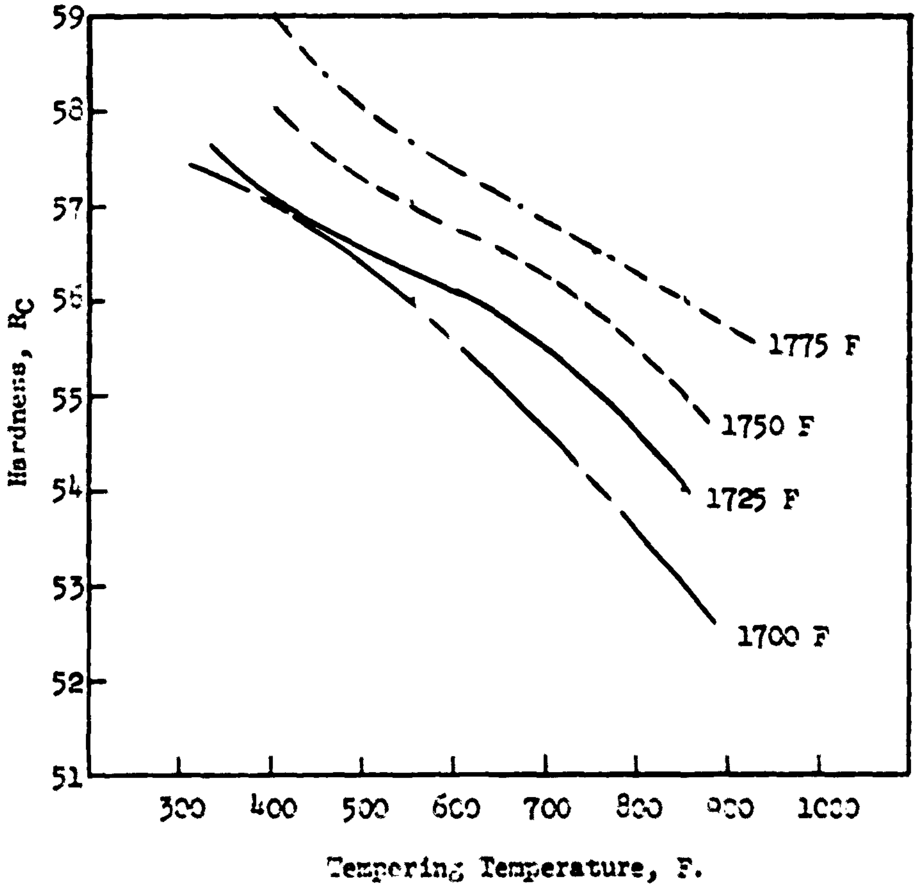
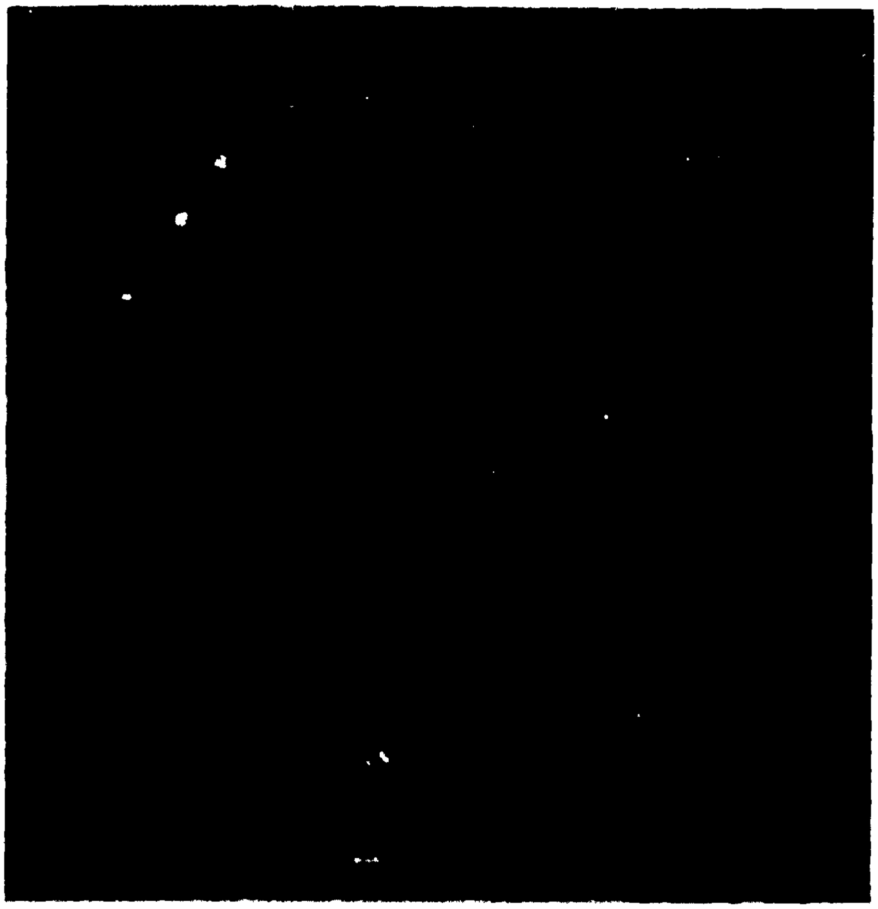


Figure 3

Effect of Austenitization Temperature on the tempered hardness of Rocoloy 270.



20,000 X

Figure 4

Electron Micrograph of Rocoloy 270
in the "As Quenched" Condition.

20,000 X

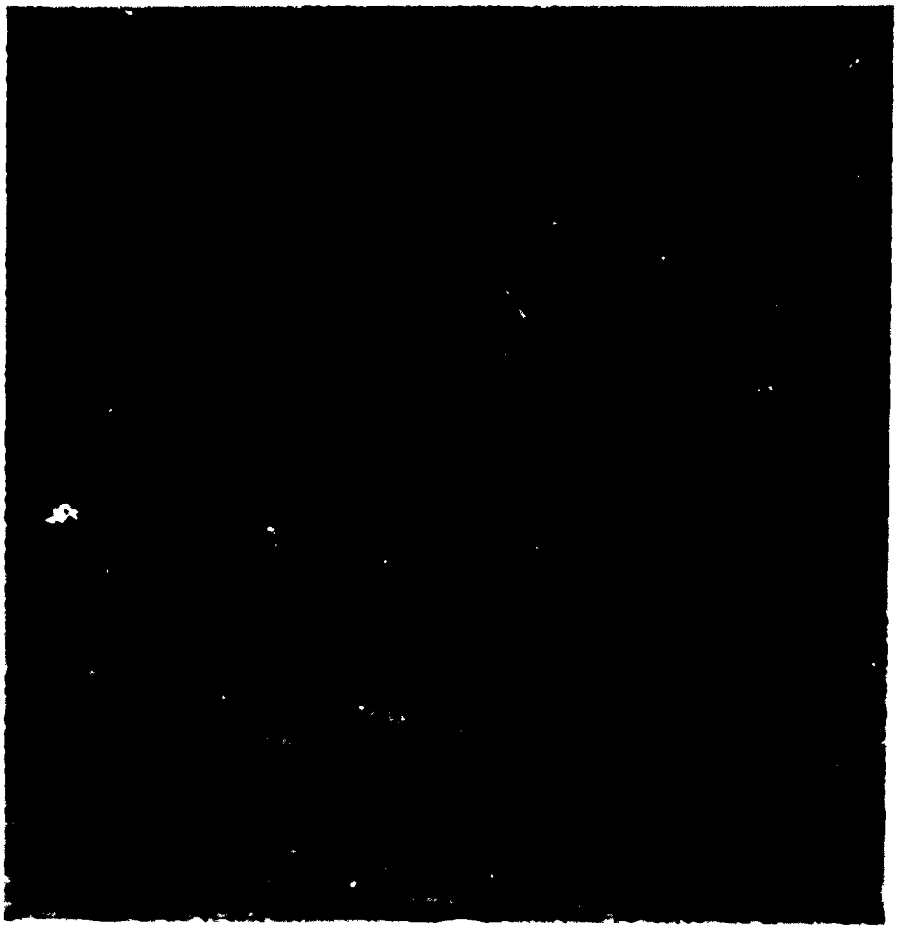
Figure 5

Electron Micrograph of Rocoloy 270
Tempered at 400 F.

20,000 X

Figure 6

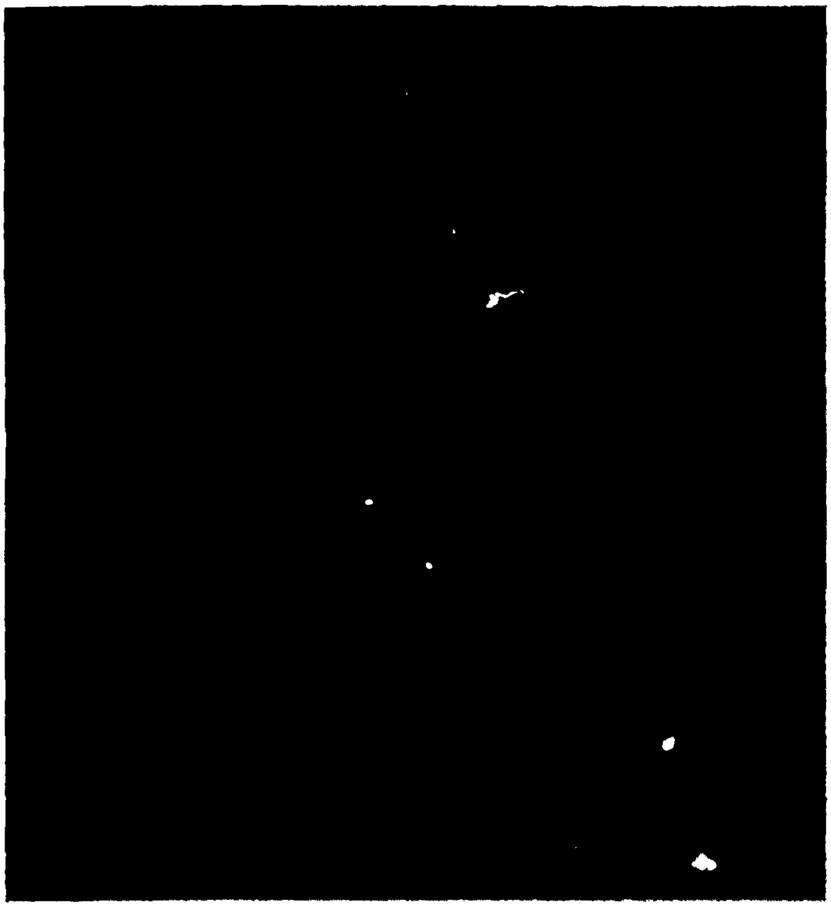
Electron Micrograph of Rocoloy 270
Tempered at 600 F.



20,000 X

Figure 7

Electron Micrograph of Rocoloy 270
Tempered at 600 F.



20,000 X

Figure 8

Electron Micrograph of Rocoloy 270
Tempered at 1000 F.

can be studied from the electron micrographs. Comparison of "as quenched" condition and 400 F tempered microstructures indicate primary grain boundaries, precipitation planes within the grain and also sub-grain boundaries. At 600 F temper, (Figure 6) the precipitation planes are more strongly etched, as are the sub-boundaries. Efforts made to connect this etching effect with the precipitation of ϵ -carbide were unsuccessful because of the diffuse electron diffraction patterns obtained from the extracted replicas.

Specimen tempered at 800 F (Figure 7) showed clear evidence of carbide agglomeration at grain boundaries and precipitation planes. This is also confirmed from the large amounts of feathery carbides seen released on the extraction replica possibly signalling third stage tempering and precipitation of cementite. At 1000 F (Figure 8) agglomeration has advanced giving discontinuous carbide masses. The extraction replica, however, revealed great deal of acicular carbide of very small size. These are yet to be identified.

Rough measurements of the electron microstructures indicated average ferrite grain diameter of 0.5 microns for the four tempering temperatures (400 F, 600 F, 800 F, and 1000 F). Also, estimates made of the mean free ferrite path vary from 1.8×10^3 A° (400 F temper) to 3.58×10^3 A° (1000 F, temper).

MECHANICAL PROPERTIES OF ROCOLOY 270

Uniaxial Tensile Properties

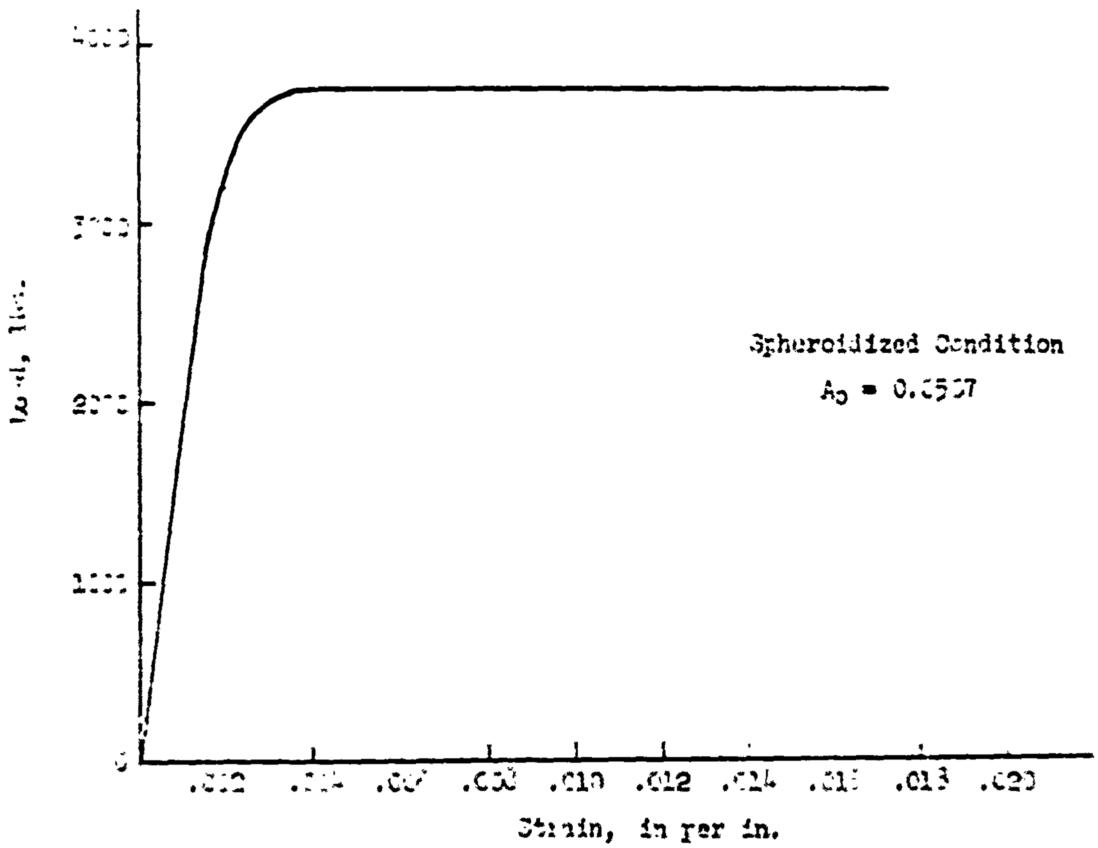
Typical tensile properties of Rocoloy 270 in sheet and bar forms in annealed condition are given in Table I. A load versus strain curve for spheroidized annealed condition is as shown in Figure 9.

Tensile properties of Rocoloy 270 sheet and bars oil quenched or air cooled and tempered at 100 F interval in the range 400 F through 1000 F are given in Table II and are also graphically presented in Figure 10.

TABLE I

Typical Tensile Properties of Rocoloy 270
in Annealed Condition (Sheet and Bar)

.2% Offset Yield Strength	73 ksi
Tensile Strength	106 ksi
Fracture Strength	165 ksi
Elongation 1"	32 Per Cent
Per Cent 2"	22 Per Cent
Reduction Area	46 Per Cent
Hardness	94 R_B



<u>Figure 9</u>

Typical Load vs Strain Curve for Rocoloy 270.

(Spheroidized Annealed Condition)

TABLE II

Room Temperature Tensile Properties[a] of Heat Treated[b] R270[c]

Section	Tempering Temperature F	.2% offset Yield Strength ksi	Ultimate Tensile Strength ksi	Fracture Strength ksi	Elongation Per Cent 1"	Elongation Per Cent 2"	Reduction in Area Per Cent	Hardness R_C
Flat (.500" x .100")	400	240	332	370	10.0	7.0	17.5	58.5
	500	266	324	373	8.3	5.3	17.0	58
	600	270	320	378	9.3	5.5	20.4	51.5
	700	265	308	361	8.6	5.3	20.5	56
	800	226	283	332	10.6	6.8	21.3	54
	900	214[2]	278	320	12.0	7.8	20.0	53
	1000	232	266	303	11.0	7.3	21.0	52
Flat[d] (.500" x .192")	550	253	302	353	11.0	6.0	21.0	53
Round (.250" Dia.)	600	268[2]	316	353	9.0	6.0	17.3	57
Round (.505" Dia.)	500	264[2]	319	-	11.0	5.0	—	58
	600	269[2]	315	374	12.0	5.0	25.0	57

a. Average of three tests except when indicated otherwise.

b. Austenitized at 1730 F for 25 min., oil quenched and triple tempered for 1 1/2 hours at the indicated temperature.

c. Representative of several heats.

d. Lower carbon heat (0.37C)

2. Refer to a.

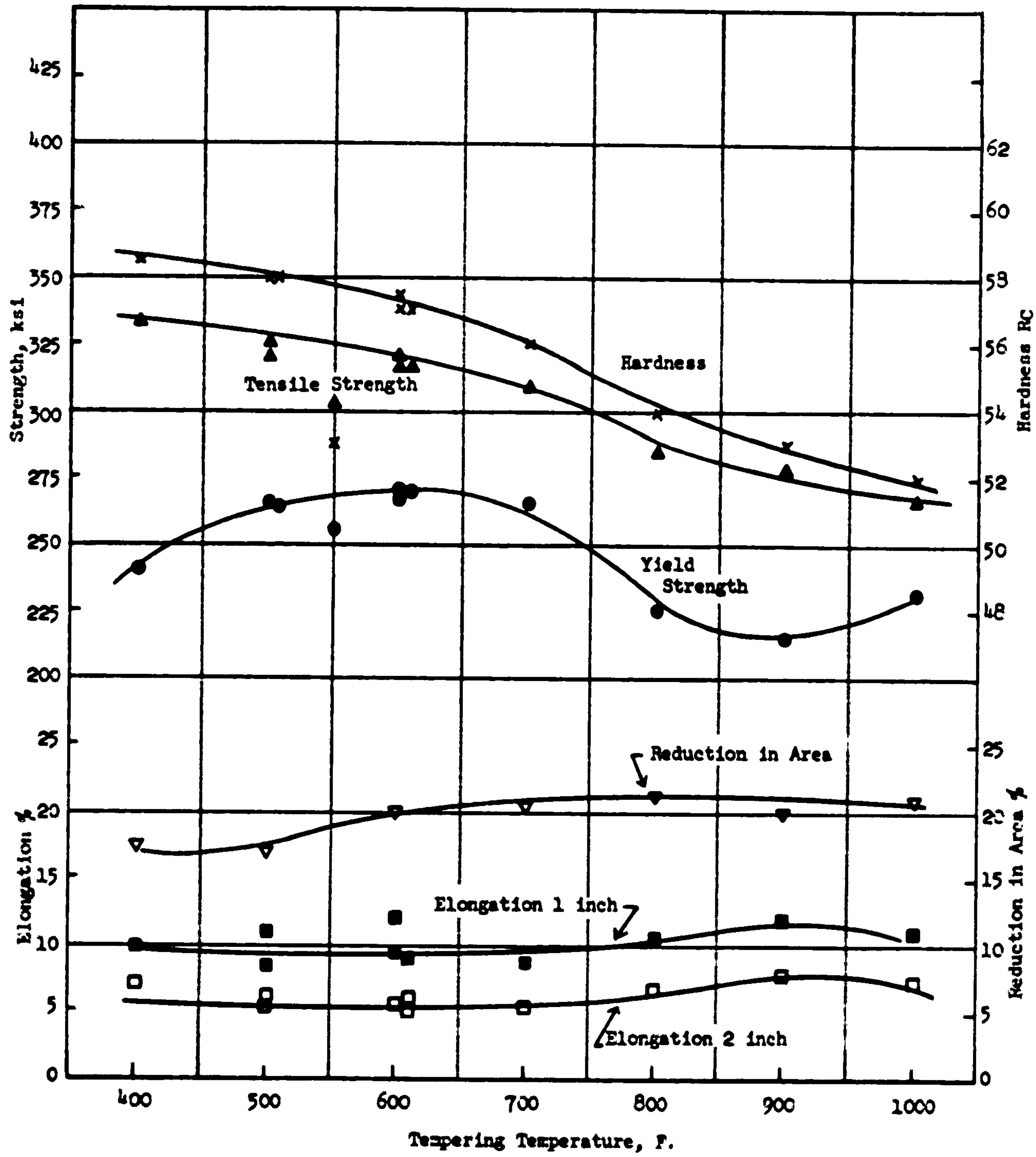


Figure 10 - Sheet Tensile Properties of Rocoloy 270, Quenched from 1730 F, and Tempered at Indicated Temperatures.

These tests have also indicated no apparent directionality of properties in cross-rolled sheets or plates. Tension test specimen configurations used in this study are given in Appendix I.

A typical load versus strain curve obtained for Rocoloy 270, hardened, and double tempered at 600 F is as plotted in Figure 11.

Elevated Temperature Properties

Elevated temperature strength has been determined for Rocoloy 270, hardened and tempered at 600 F. Data summarized in Table III indicates excellent elevated temperature load carrying capabilities of this steel containing not more than about 7 1/2 per cent total alloying constituents.

Notch Tensile Properties

Notch tensile strength of Rocoloy 270, for several tempered conditions, has been studied by comparing tension test data obtained using edge-notched specimens having $K_t = 5$, 12.5, and 17, with those for unnotched specimens. The notches reduced the section width of an unnotched specimen by 30 per cent. Specimen dimensions, notch-geometry and other pertinent details can be found in Figure 12. Notch tensile test data obtained are summarized in Table IV.

Edge-notch tensile tests on Rocoloy 270 specimens with $K_t = 5$ and $K_t = 17$ were made on air melt and vacuum consumable arc remelted portions of a 15,000 lb. electric arc furnace heat. However, the carbon and chromium contents of this heat fell outside the chemical limits established for Rocoloy 270. The carbon of this heat ranged from 0.49 to 0.52 per cent. The edge-notch test data, therefore, indicate some notch sensitivity even with engineering notches and appreciable sensitivity in the presence of very sharp notches.

The notch-tensile work with $K_t = 12.5$ was done on specimens from a

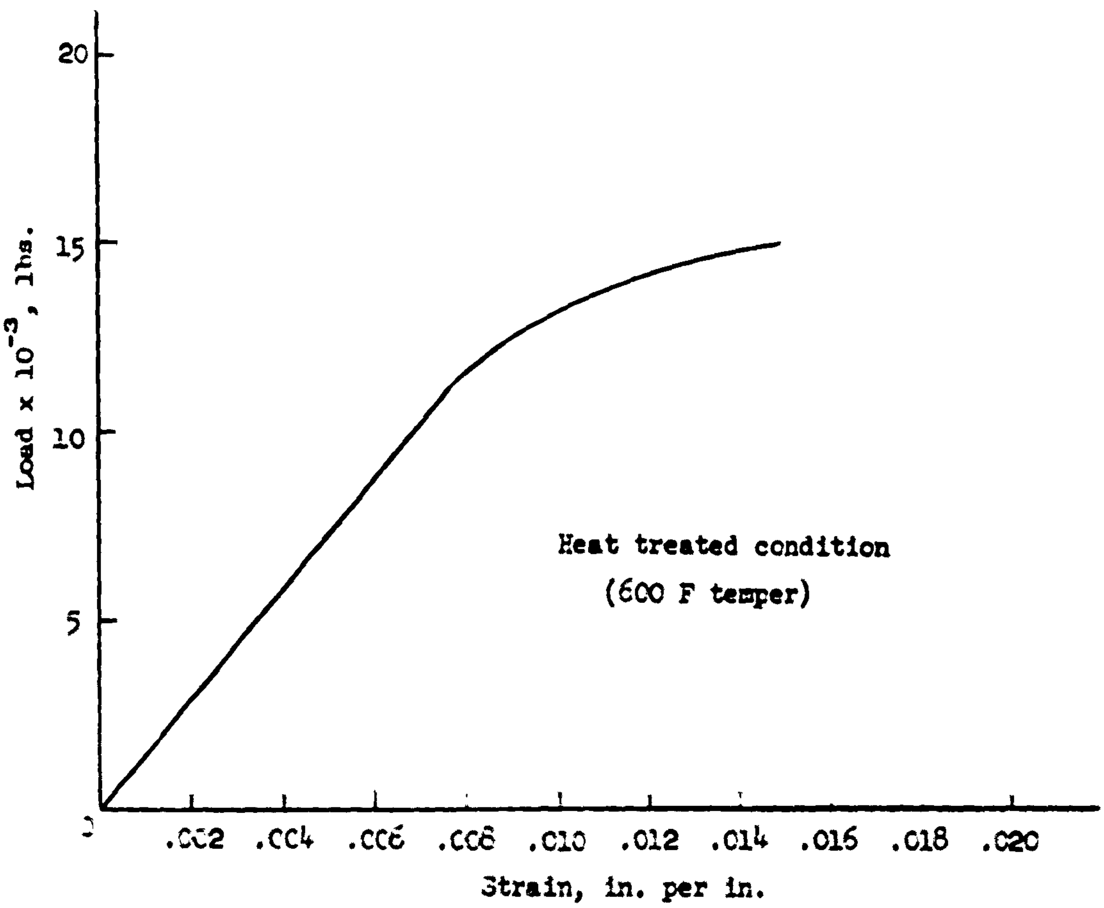


Figure 11

Typical Load vs Strain Curve for Rocoloy 270.
(Hardened and tempered at 600 F)

TABLE III

Elevated Temperature Tensile Properties of Rocoloy 270[1]

(0.090 in. Thick Sheet Specimens Austenitized at 1730 F, Quenched in Oil and Subsequently Tempered at 600 F)

Testing Temperature F	Average[2] 0.2% offset Yield Strength ksi	Average Ultimate Tensile Strength ksi	Elongation in 2 in. Per Cent	Modulus of Elasticity
200	257	313	7.5	27.4
300	251	310	7.5	29.2
400	241	309	8.5	26.7
500	229	285	9.6	25.4
600	218	263	11.8	24.2

1. Carbon of this heat was 0.41 to 0.43 per cent. Chromium 1.50 per cent.

2. Average value of two tests.

Figure 12

Center Notch Fracture Toughness Test Specimens

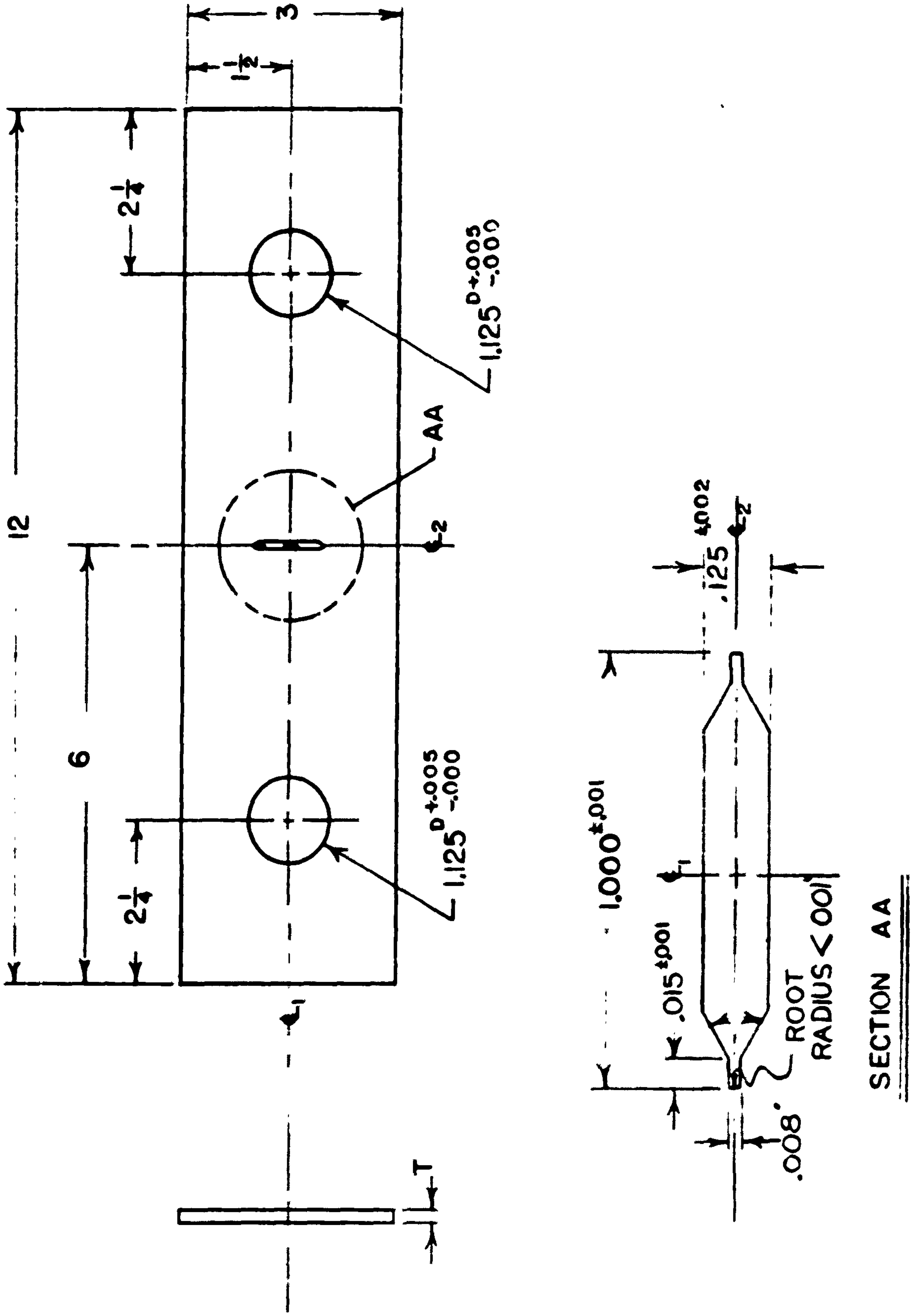


TABLE IV

Edge Notch Tensile Test Results for Rocoloy 270[a]

Tempering Temperature F	Notched/Unnotched Tensile Ratio[b c]					Unnotched Ultimate Tensile Strength ksi
	Air Melt $K_t = 5$	Consumable Arc Remelt $K_t = 5$	Air Melt $K_t = 12.5$	Air Melt $K_t = 17$	Consumable Arc Remelt $K_t = 17$	
400	0.675	0.896	0.75	0.358	0.271	332
500	0.925	0.936	0.80	0.199	0.241	324
600	0.913	0.978	0.87	0.233	0.208	320
700	0.842	0.971	0.72	0.195	0.236	308
800	0.888	0.880	0.80	0.251	0.250	283
900	0.888	0.962	--	0.302	0.290	278
1000	0.953	1.020	--	0.480	0.545	266

a. Austenitized at 1730 F for 25 min., oil quenched and triple tempered at indicated temperatures.

b. All specimens have a 30% V-Notch.

c. The specimens for $K_t = 5$ and 17 are from a high carbon heat of Rocoloy 270.

2000 lb. electric arc furnace melted heat having carbon around 0.41 per cent and the data obtained indicate that Rocoloy 270 is only slightly sensitive to the presence of sharp notches.

It is concluded from this work that Rocoloy 270 of optimum chemical analysis should exhibit very slight or no notch sensitivity in the presence of engineering notches ($K_t = 3$ to $K_t = 6.5$) and some degree of sensitivity increasing with the notch acuity.

Fracture Toughness

Fracture toughness evaluation of Rocoloy 270 (0.49 to 0.52 carbon heat) has been made using 3 inch wide center-notched sheet specimens. The notches were inserted by manual sawing with a fine jeweler's saw. A drawing of these specimens is shown in Appendix II. Heat treating of the specimens was done subsequent to notching, in freshly deoxidized neutral molten salt baths. Data of these tests are as shown in Table V. The low G_c values obtained appeared incompatible with good biaxial ductility exhibited by this material under hydrostatic cup tests. A study was therefore made of the effect of marking fluids on the delineation of the slow crack growth. The results of this study is presented as a bar chart in Figure 13. It can be clearly seen from this chart that use of certain marking fluids can lead to erroneous G_c and K_c values. The reasons for this behavior are not presently known. Design engineers are urged to excercise caution in regarding G_c values as a criterion for material selection without due consideration of the suitability of crack growth testing procedure for ultrahigh-strength materials.

Impact Strength

Impact strength of Rocoloy 270 was studied using conventional V-notched Charpy specimens. Data of this study are given in Table VI and the effect of melting technique (quality) and tempering temperature on impact

TABLE V

Fracture Toughness Test[a] Results for Rocoloy 270

Tempering Temperature F [b]	G_c ipsi [c]	K_c ksi/$\sqrt{in.}$ [c]	Net Section Stress
400	562	130	148
500	504	123	140
600	456	117	130
700	404	110	126
800	410	111	128

(a) Center notched specimens conforming to ASTM specifications.

(b) Austenitized at 1730 F for 25 min., oil quenched and triple tempered as indicated.

(c) $G_c = \frac{\sigma^2 W}{E} \quad \text{Tan} \frac{\pi a}{W} \qquad K_c = \sqrt{E\, G_c}$

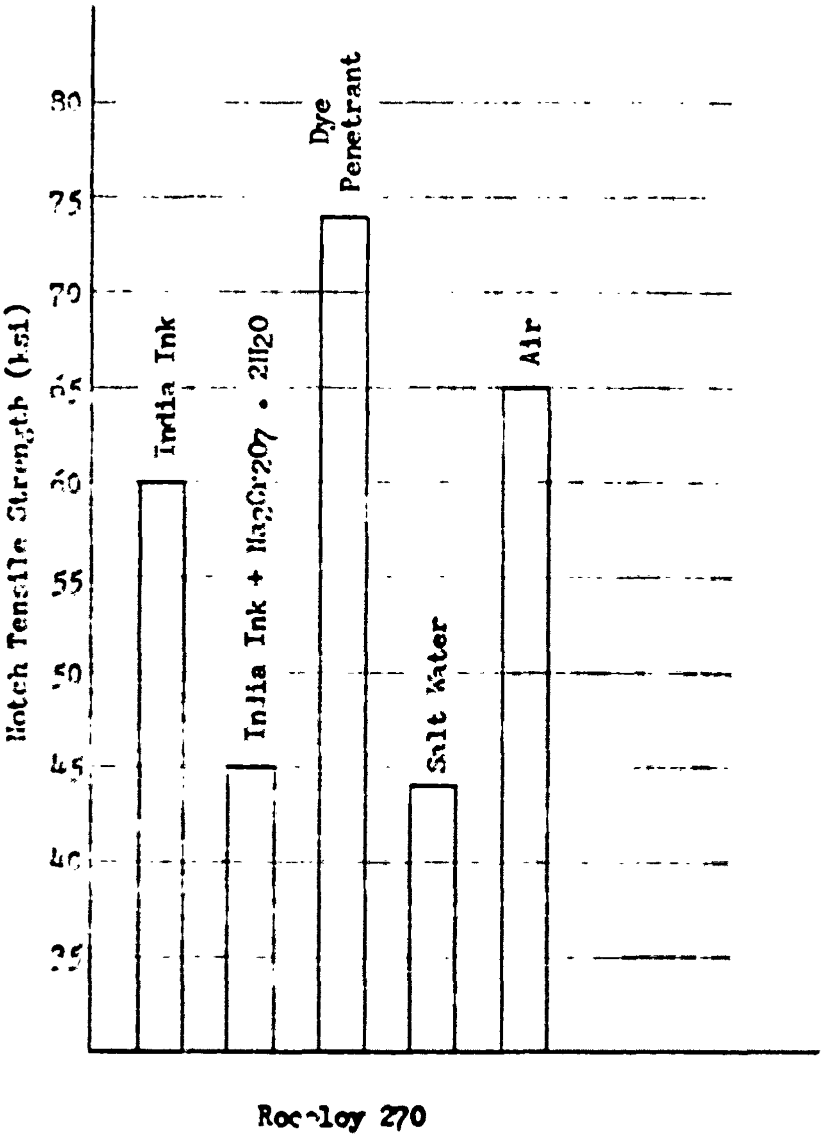


Figure 13

Influence of Marking Fluids on G_c
(Fracture Toughness Value) of Rocoloy 270.

TABLE VI

Charpy Impact Test Results for Rocoloy 270[a] and Data on the Effect of Melting Variables on Impact Toughness

LONGITUDINAL DIRECTION[b]

(0.49 to 0.52 C Heat)

Charpy, ft. lbs.

Melting Method

Tempering Temperature F	AM	VCA	DVCA	VI
400	14-16	12-14	11-12	15-16
500	14-16	12	12-14	14
600	11-12	11-12	12-14	14
700	9-10	9	9-10	10-12
800	10-13	9-12	9	11-13
900	12-14	12-14	12-13	12-12
1200	27	29	32	26
TRANSVERSE DIRECTION[c]				
600	10-13	14-16	15-18	13-15

(a) Austenitized at 1730 F.

(b) Specimens taken from 1 1/4" dia. bar stock.

(c) Specimens taken from 4" x 4" billet stock.

AM - Air melt.

VCA - Vacuum consumable arc remelt.

DVCA - Double vacuum consumable arc remelt.

VI - Vacuum induction melt.

strength is graphically presented in Figure 14. The room temperature impact strength of Rocoloy 270 is significantly higher than steels of considerably lower tensile strength. If still higher low-temperature impact strength is desired, omission of tungsten and increasing the nickel content approximately 0.40 per cent above the top limit in the composition is recommended. The effect of testing temperature on the impact properties of vacuum melted Rocoloy 270 was studied and the results obtainad are presented as an impact transition curve in Figure 15.

Fatigue Properties

Rotating beam fatigue and sheet bending fatigue tests have been conducted on Rocoloy 270 specimens hardened and tempered at 600 F. S-N curves shown in Figure 16 have been developed for Rocoloy 270 melted in air, and under vacuum by several improved methods. Double consumable arc remelted material exhibits the best fatigue properties both in terms of load carrying capability and the endurance limit. Rocoloy 270 exhibits considerably superior fatigue strength compared with AISI 4340 or H-11 as indicated by generalized S-N curves presented in Figure 17.

Fatigue properties of Rocoloy 270 determined as a function of austenitizing temperature indicated that non-optimum austenitizing treatment results in lowered fatigue strength. Results of this study are presented in Figure 18.

Stress-Rupture Properties

Stress-rupture properties of Rocoloy 270 was investigated in the temperature range 400 F through 700 F. Specimens for these tests were heat

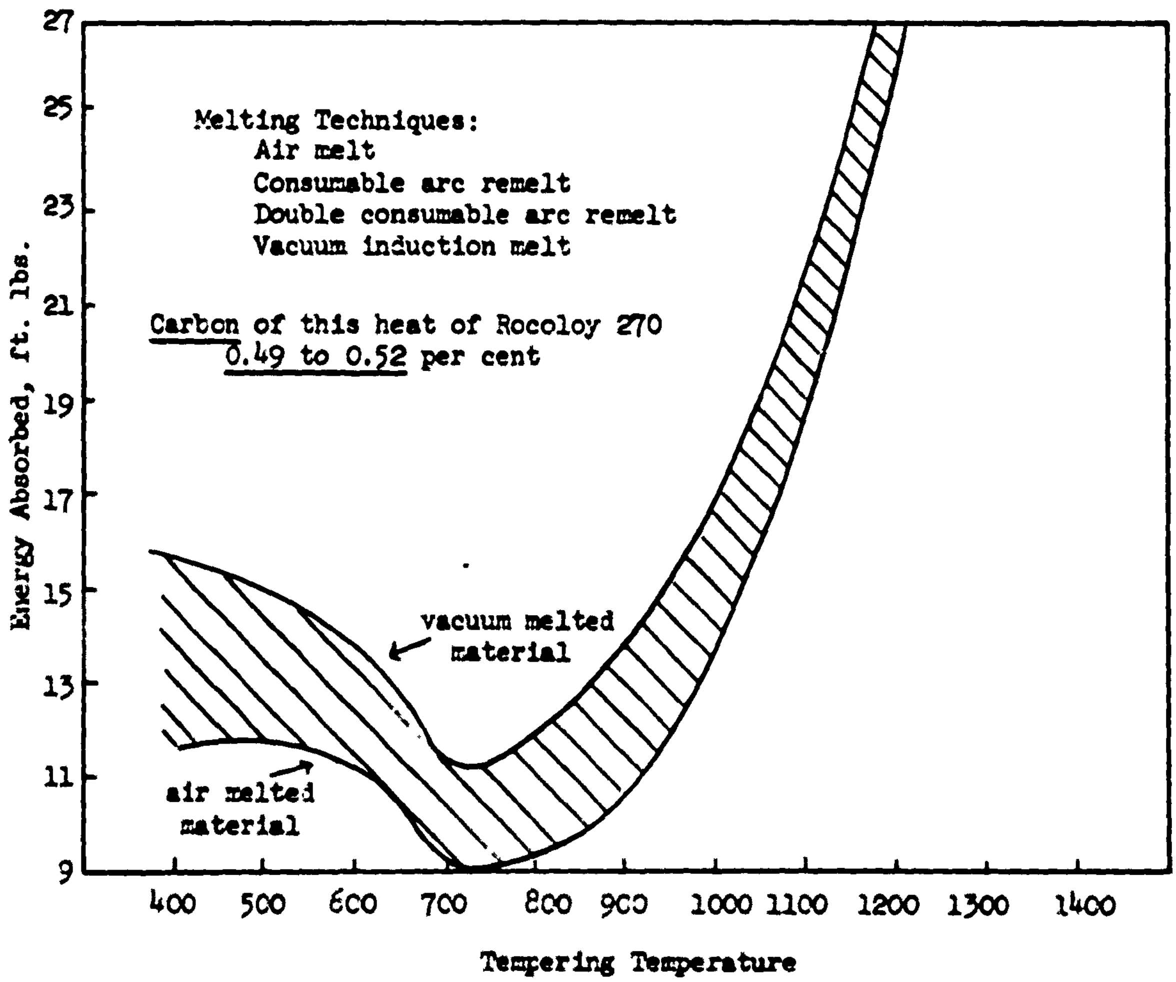


Figure 14

Effect of Tempering Temperature on the Impact Strength of Rocoloy 270 melted by various techniques.

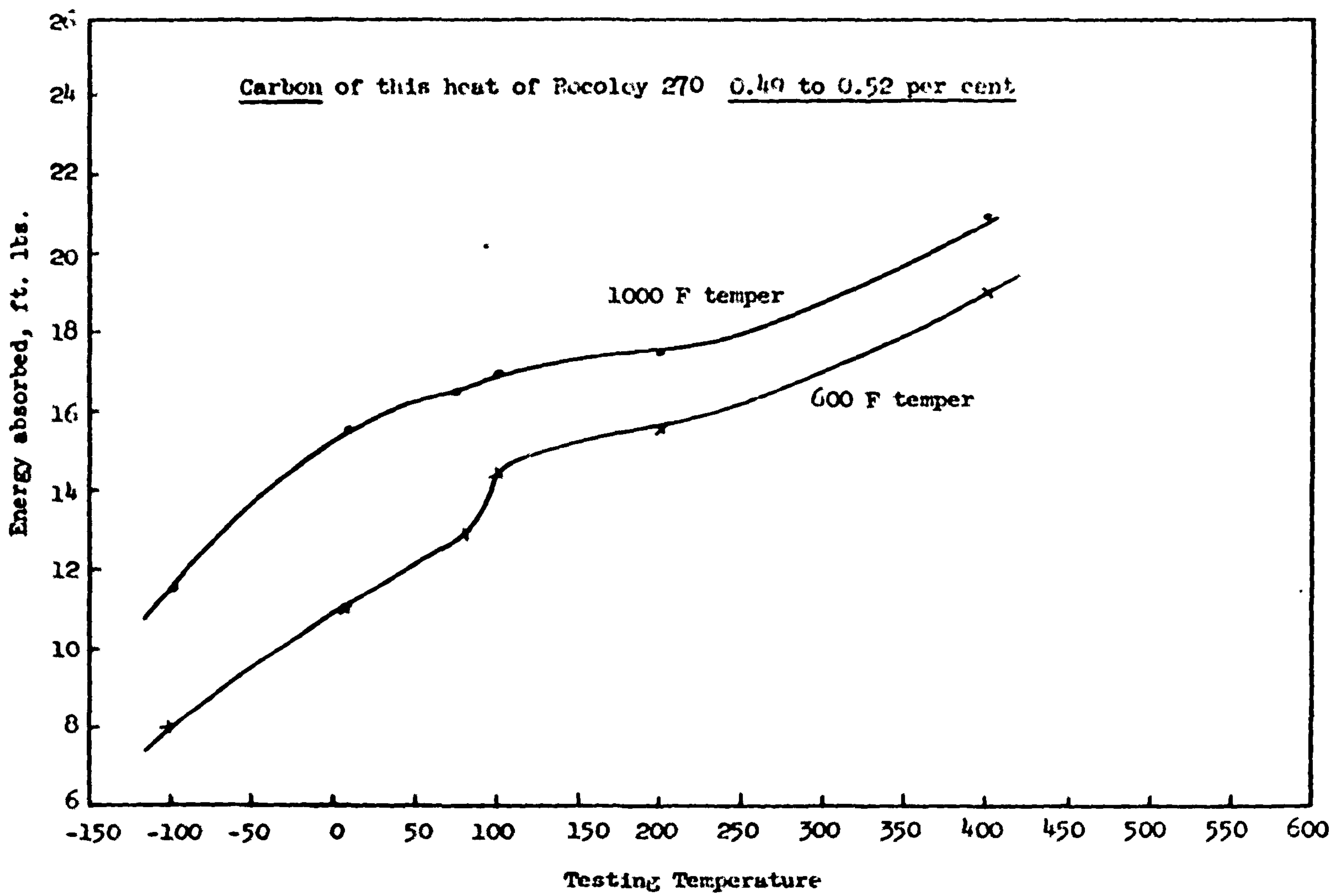


Figure 15 - Effect of testing temperature on the Charpy impact strength of Rocoloy 270.

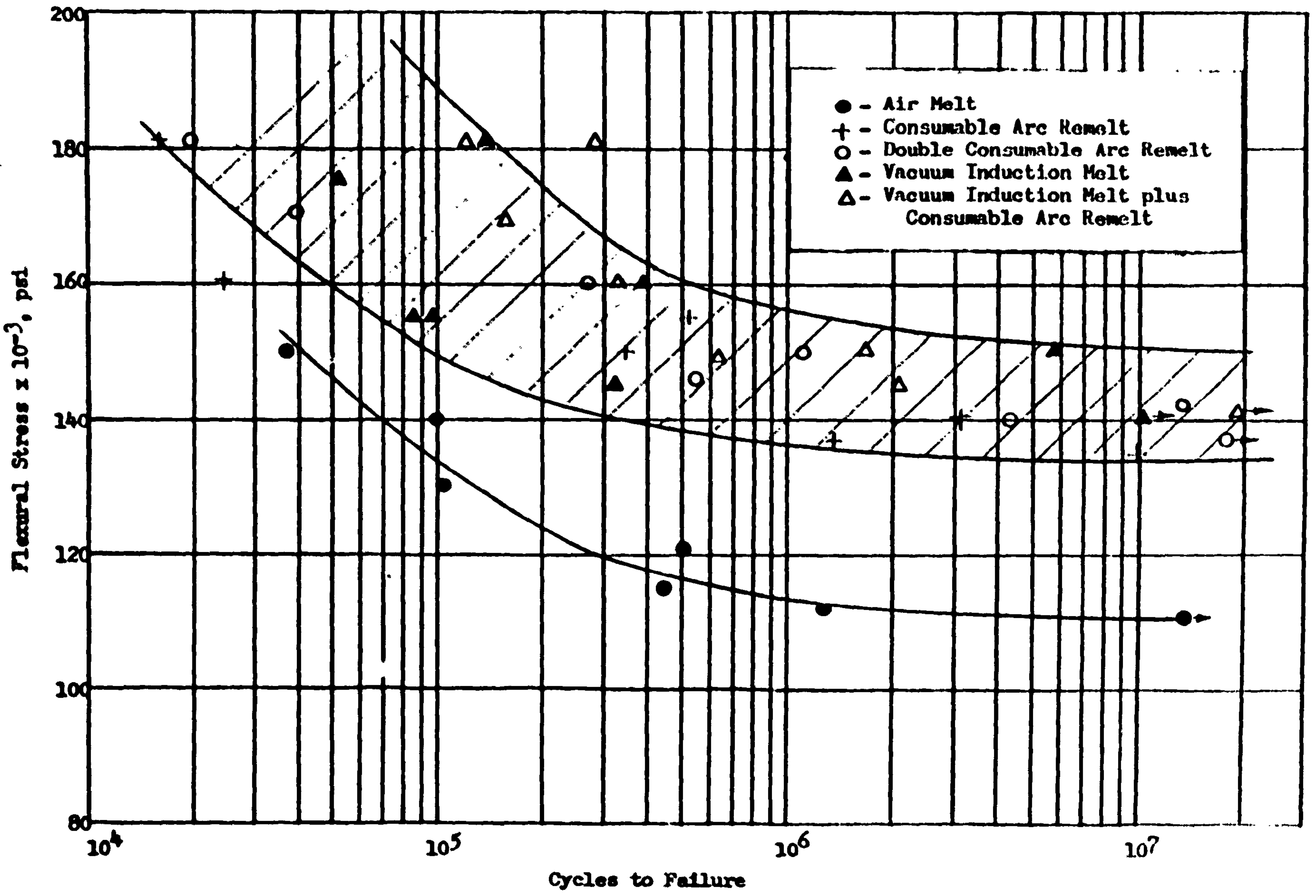
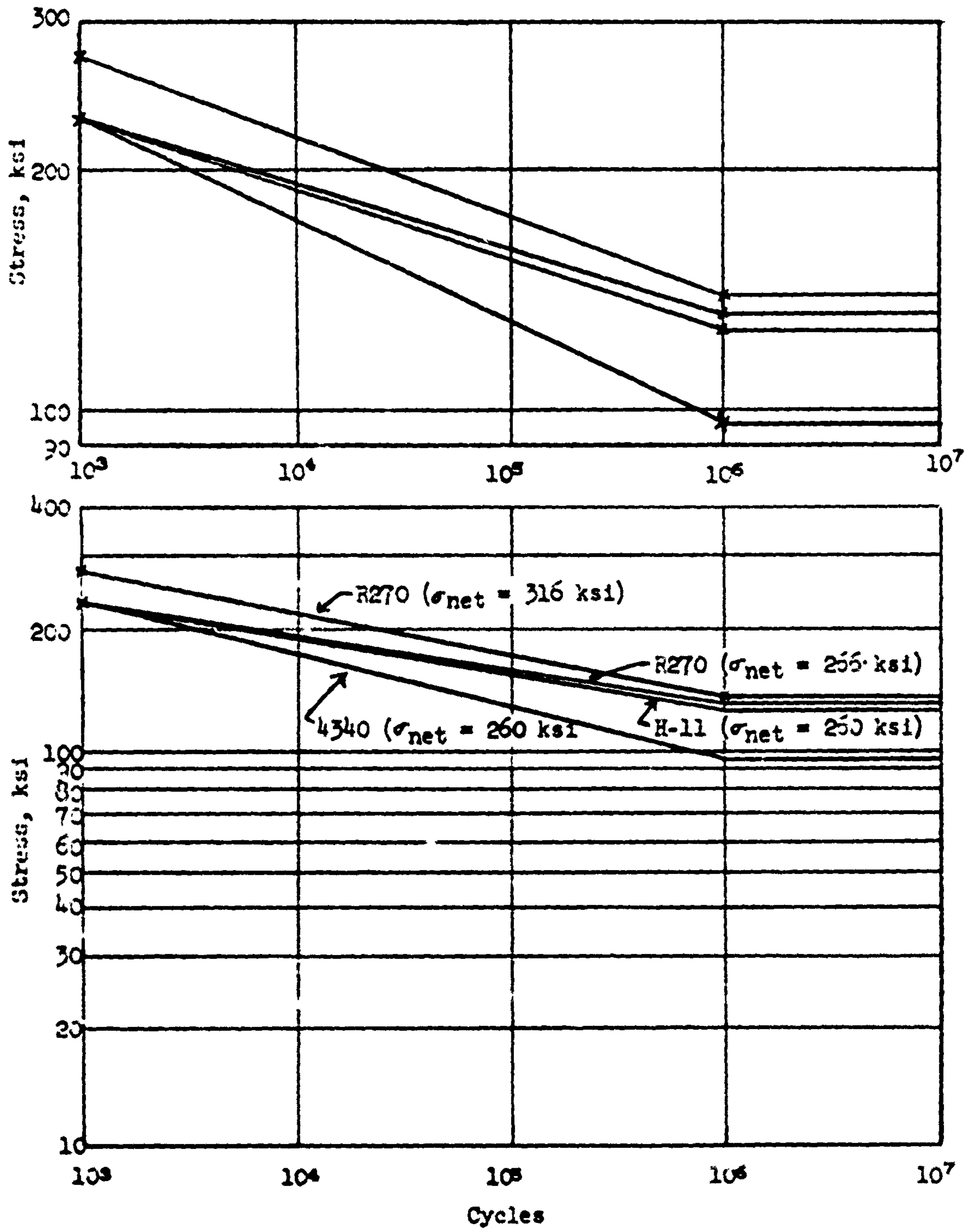


Figure 16 - Effect of Melting Variable on Fatigue Characteristics of Rocoloy 270 Heat Treated to 268,000 psi 0.2 per cent offset Yield Strength and 320,000 psi Ultimate Strength Levels (Hardness R_C 56 to 57).



<u>Figure 17</u>

Generalized S-N curves showing Rocoloy 270 in Comparison with AISI 4340 and H-11.

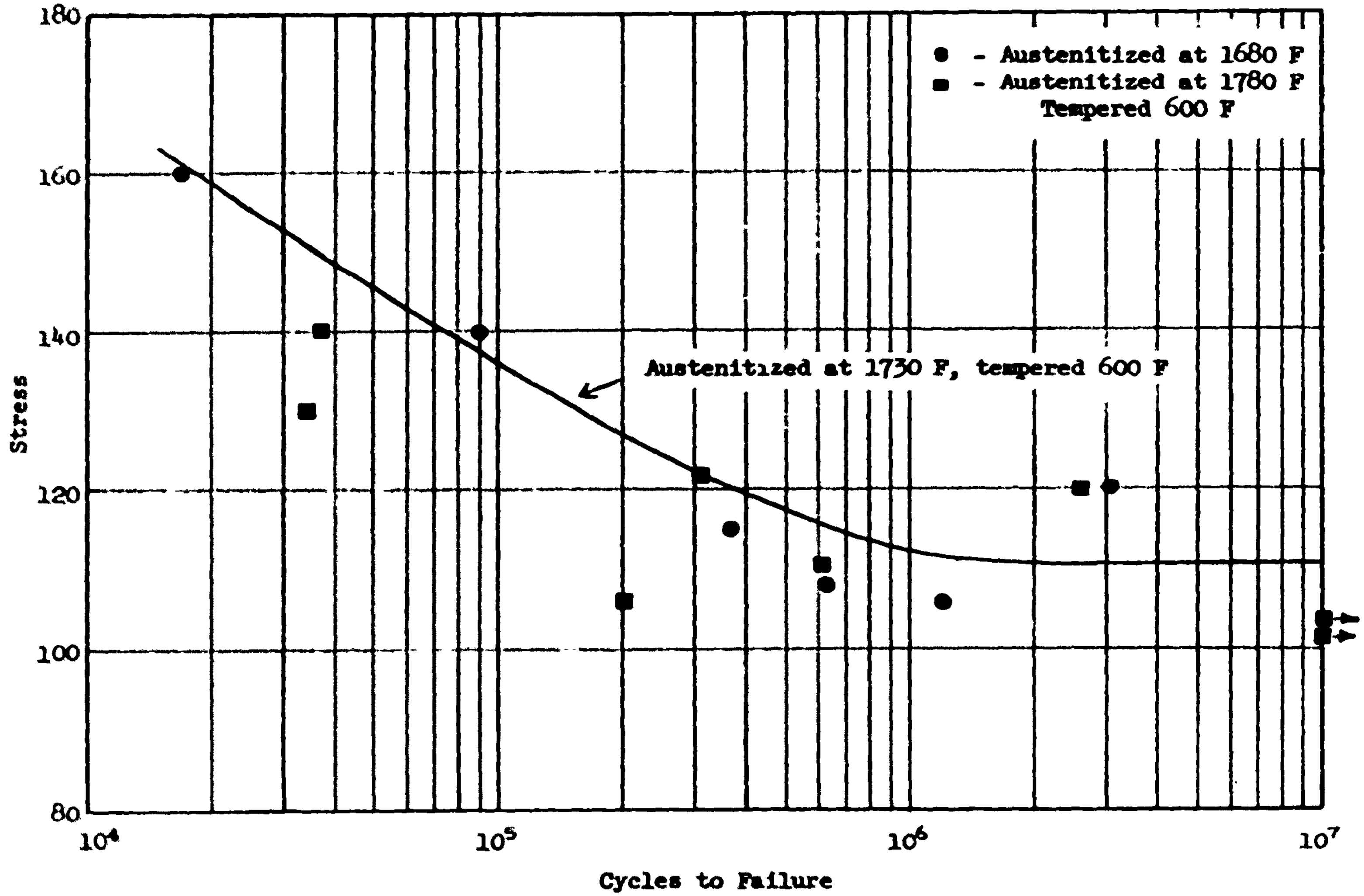


Figure 18

Effect of Austenitization Temperature on the Fatigue Properties of Rocoloy 270 (Air Melt).

treated to approximately 270,000 psi, 0.2 per cent offset yield strength and 320,000 psi ultimate strength level. The data of this work are summarized in Figure 19 in terms of time to failure at various stress level and temperatures. For a low alloy steel Rocoloy 270 exhibits remarkable stress-rupture strength at temperatures up to 600 F.

Multiaxial Strength

Multiaxial stress capabilities of Rocoloy 270 have been determined through hydrostatic pressure testing of hemispherical cups and scale model pressure containers. Figure 20 shows the cup test specimen, testing fixture and the load-strain measuring device attached to the test fixture. The scale model (3.5 in. dia. x 18 in. long) pressure vessels used are shown in Figure 21. These two types of test specimens were heat treated to various strength levels and tested for developing biaxial strength data for Rocoloy 270 to supplement the unixial tensile test results.

The biaxial strength of Rocoloy 270 as a function of a tempering temperature is presented in Figure 22. Similar results developed by hydrotesting the vessels are presented in Figure 23. Both test results indicate achievements of biaxial yield strength in excess of 270,000 psi. However, it should be emphasized that bending stresses of any magnitude cannot be sustained by Rocoloy 270 pressure vessels. As the yield strength increases above 220,000 psi extra care in designing components in order to avoid bending stresses is of paramount importance. Relative merit of Rocoloy 270 in the form of containers is clearly shown by the biaxial test results presented in Figure 24 for various high strength steels.

FORMABILITY

Forming operations performed on Rocoloy 270 including forging, rolling, deep drawing, ironing, coining, hot and cold spinning indicated no difficulties

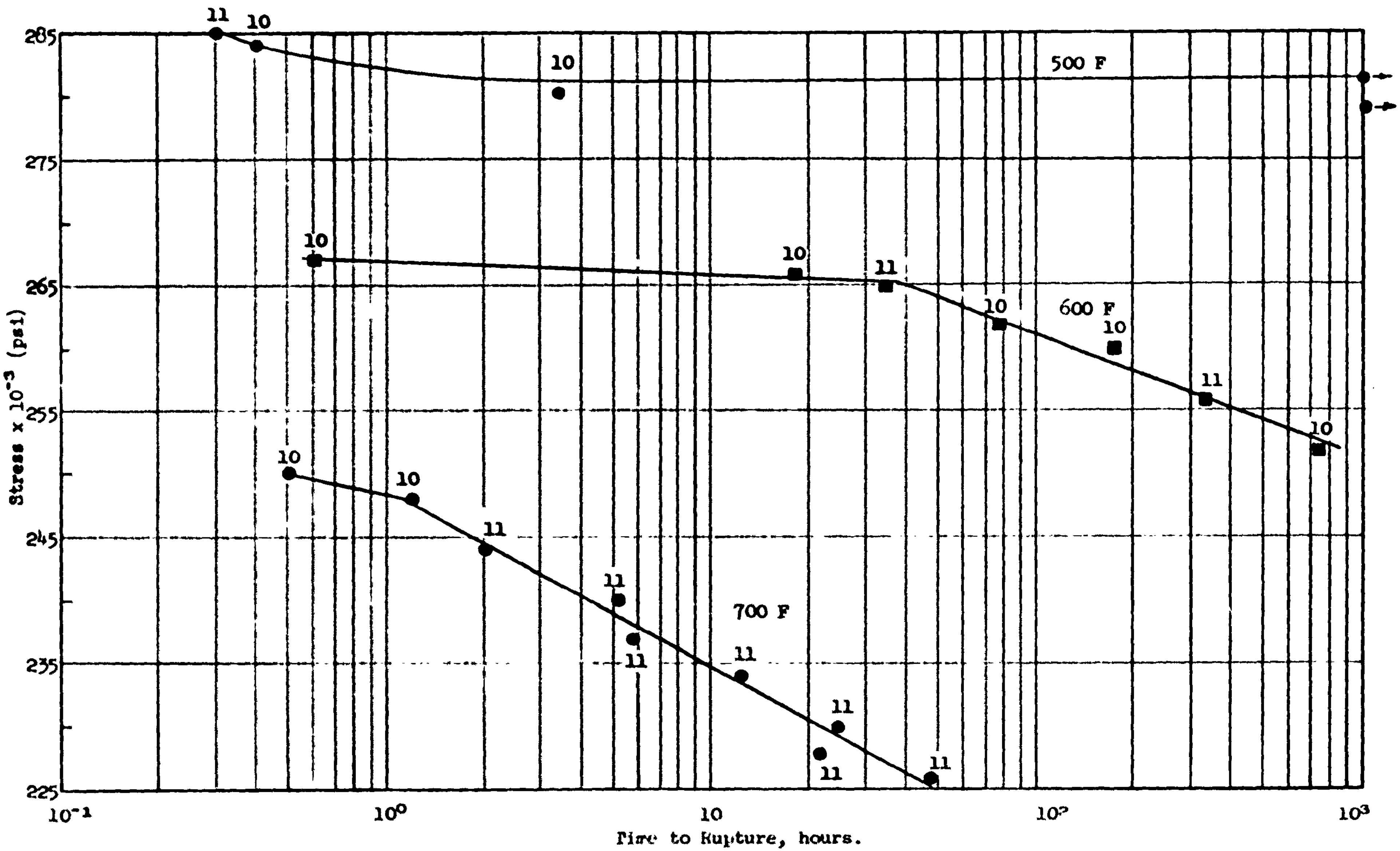


Figure 19 - Elevated Temperature Stress-Rupture Data for Hocoloy 270.

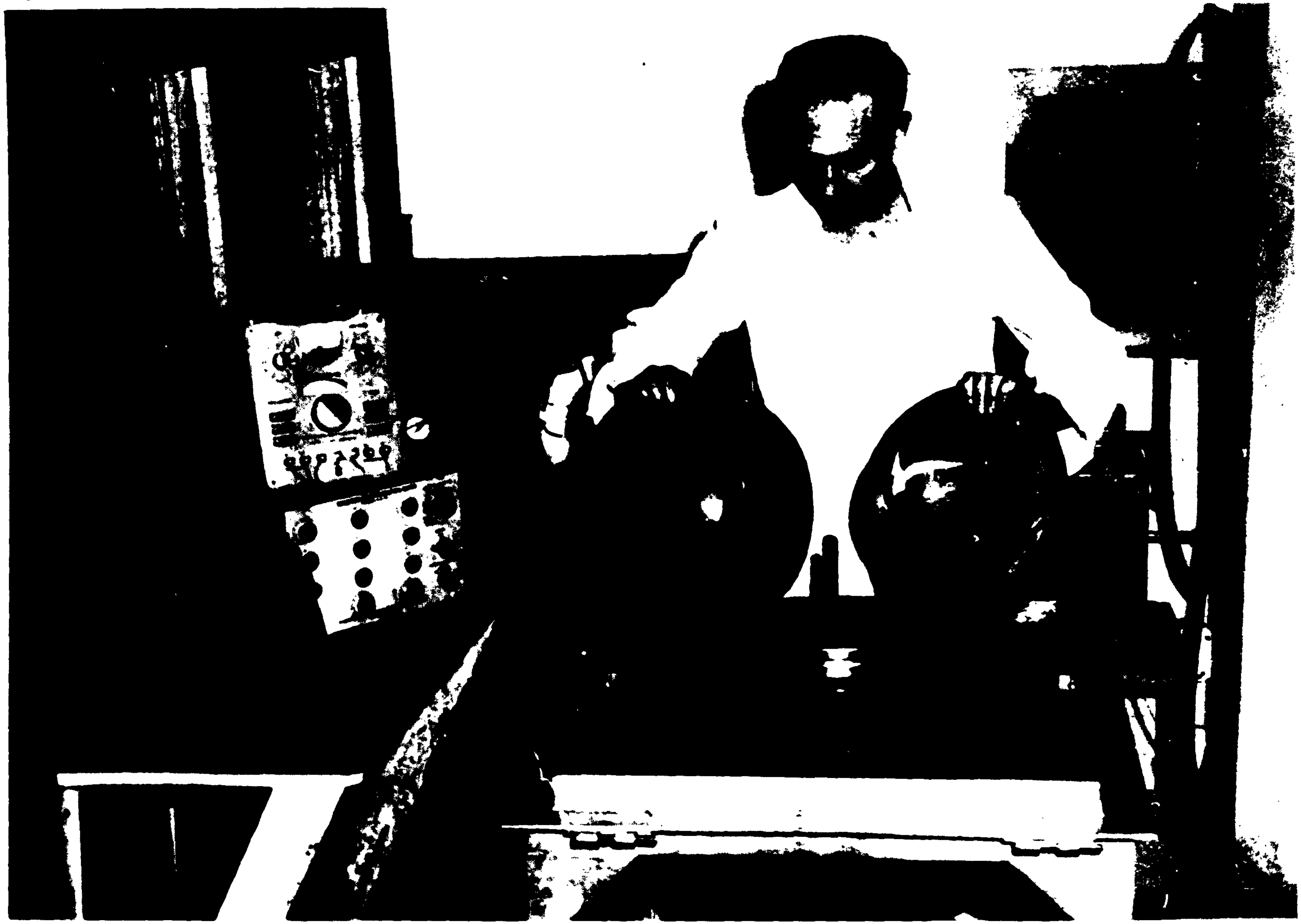
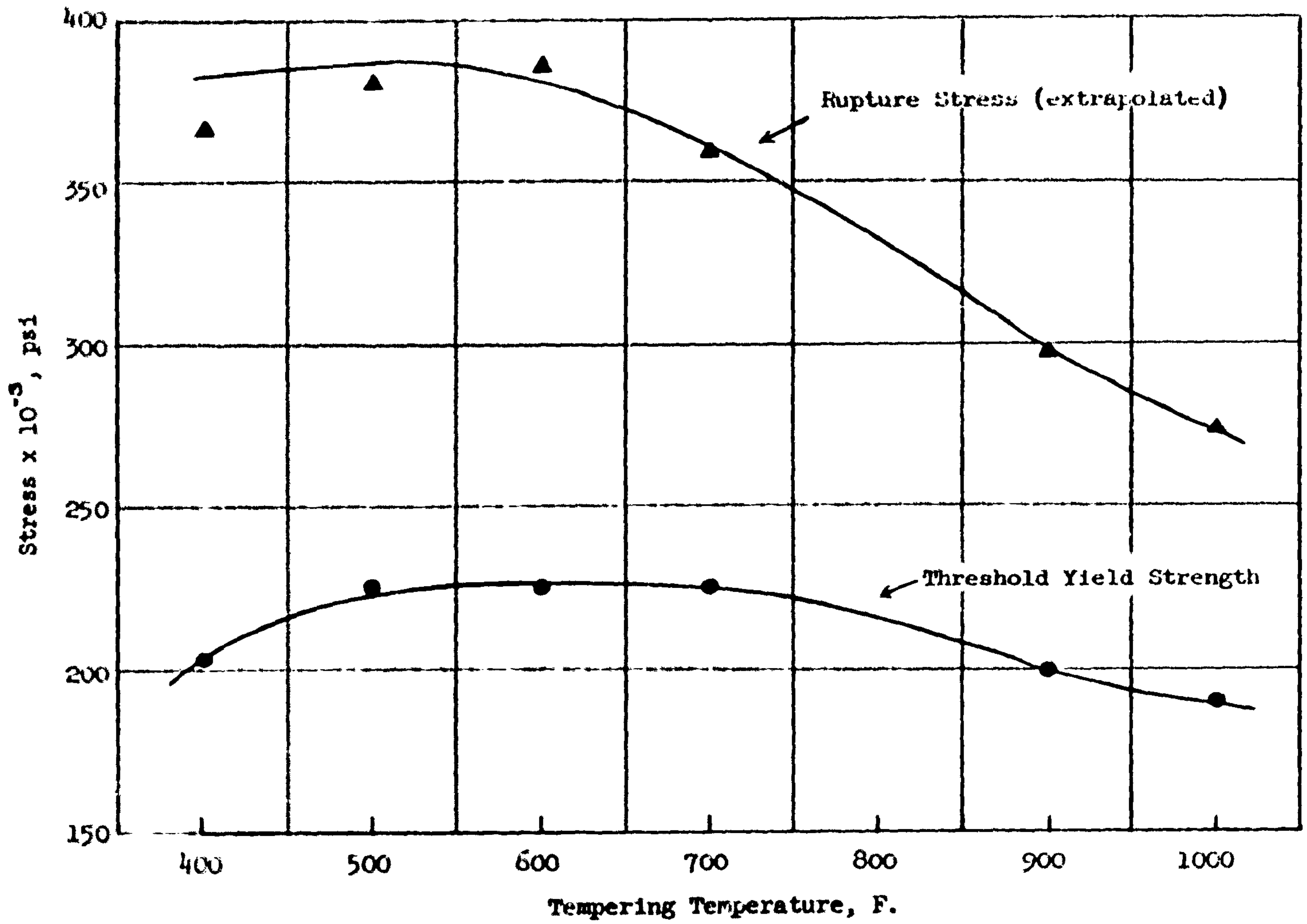


Figure 20

Rocoloy 270, 10 in. Diameter Hemispherical Cup and Biaxial Strength Evaluation Fixture.

Figure 21

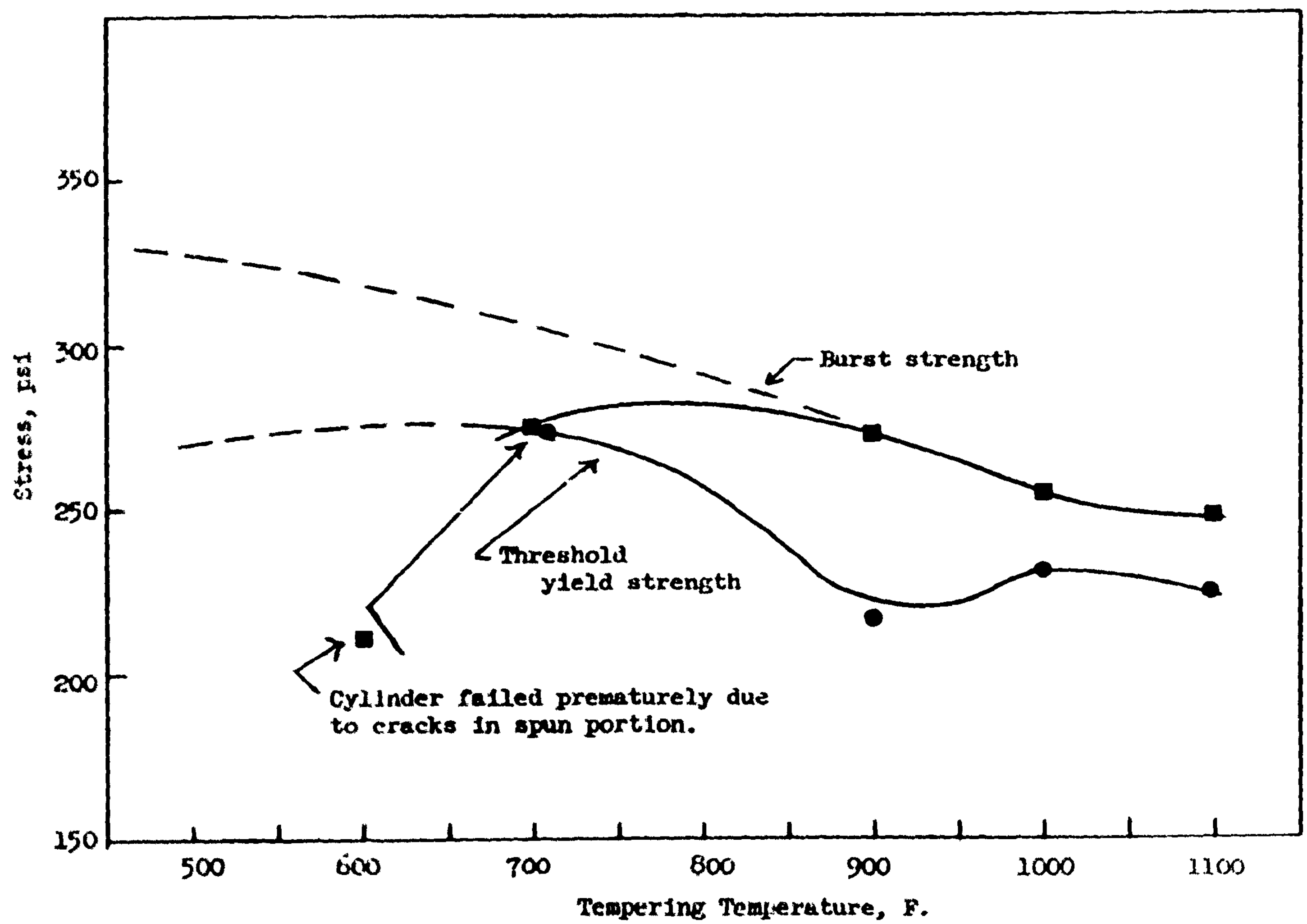
Rocoloy 270 3.5 inch Diameter Cylinders.



<u>Figure 22</u>

Biaxial Strength of Rocoloy 270 obtained by Hydrostatic Testing of Hemispherical Cups Hardened and Tempered at Indicated Temperatures.

(Material had surface decarburization to a depth of .011 inch in original material thickness .080 inch)



<u>Figure 23</u> - Results on Rocoloy 270, 3.5 in. Diameter Cylinder Hydrotests. (Cylinders austenitized at 1730 F, quenched in oil and tempered at indicated temperatures).

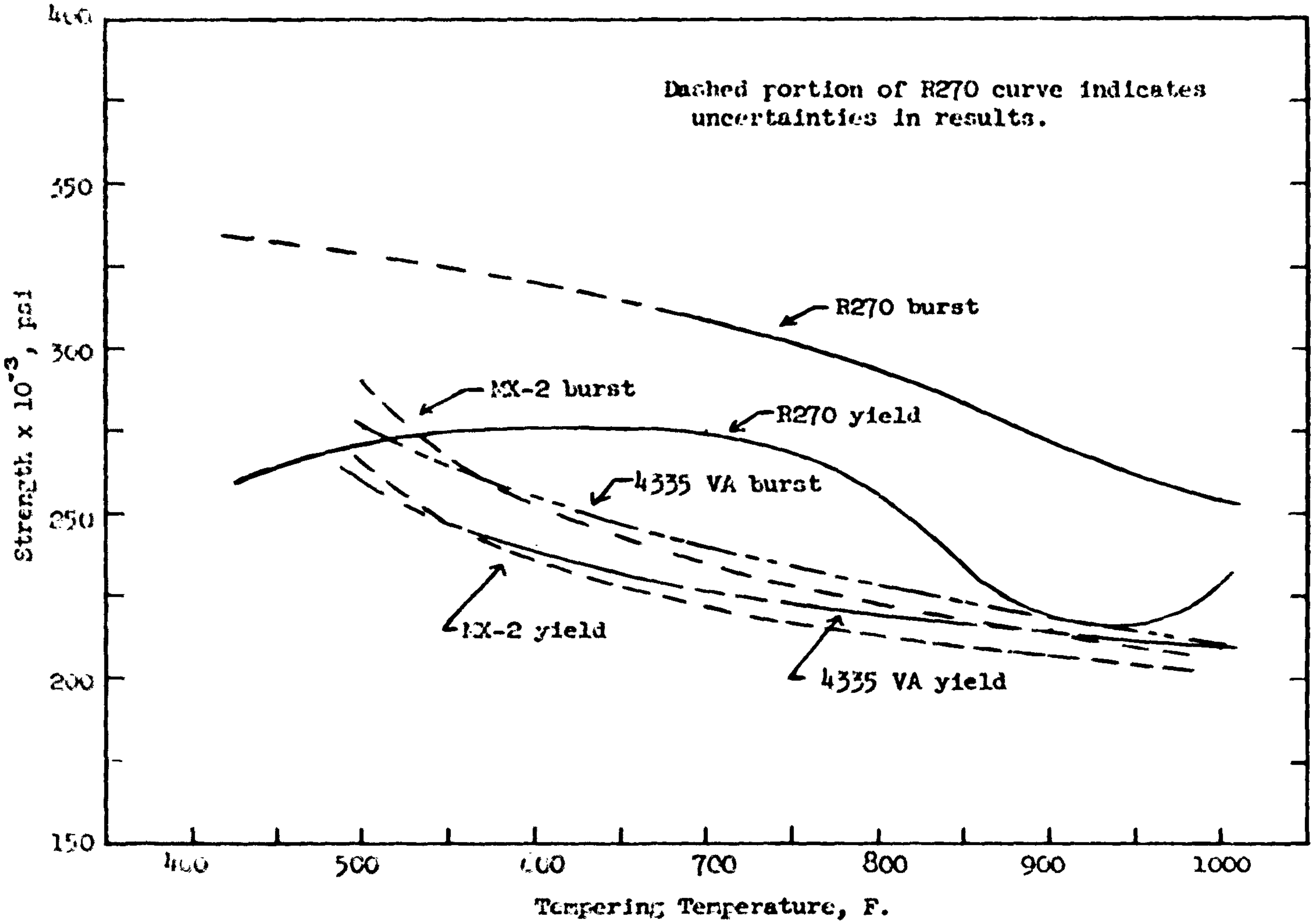


Figure 24

Biaxial Strength Data for Various Steel
showing Relative Superiority of Rocoloy 270.

or problem areas.

Rocoloy 270 may be deep drawn cold using conventional tools and procedures with individual draws not exceeding 35 per cent. Recrystallization annealing between draws can be done readily by heating to 1325 F for 10 to 15 minutes.

Hot spinning can be satisfactorily performed by heating Rocoloy 270 to a temperature range 1850 to 2050 F. No appreciable grain growth is observed upon heating up to 2125 F. Figure 25 serves to illustrate the various forming operations performed on a sheet blank of Rocoloy 270 in the course of fabricating 3.5 in. diameter containers.

WELDING

Welding research performed on Rocoloy 270 is limited to tungsten arc inert gas shielded welding of sheets ranging in thickness from 0.030 in. to 0.160 in. The effects of varying amperage, voltage, welding speed and the use of various matched (MX-2) and overmatched (17-22 AS) filler wires on the bend ductility and tensile strength of the weld deposit were evaluated. Metallographic studies were conducted to observe the refinement of grain structure produced in the weld and heat affected zone as a result of using single and multiple weld passes.

Results of weld bend ductility studies are presented in Table VII and pictures of typical bends made on welded Rocoloy 270 are shown in Figures 26, 27, and 28.

Typical tensile test results obtained on heat treated butt welded sheet specimens of Rocoloy 270 using MX-2 and 17-22 AS weld filler wires are given in Table VIII. These results serve to indicate that invariably 100 per cent weld joint efficiency and tensile strength of the parent material has been secured in TIG welded butt joints made using both types of filler wires.

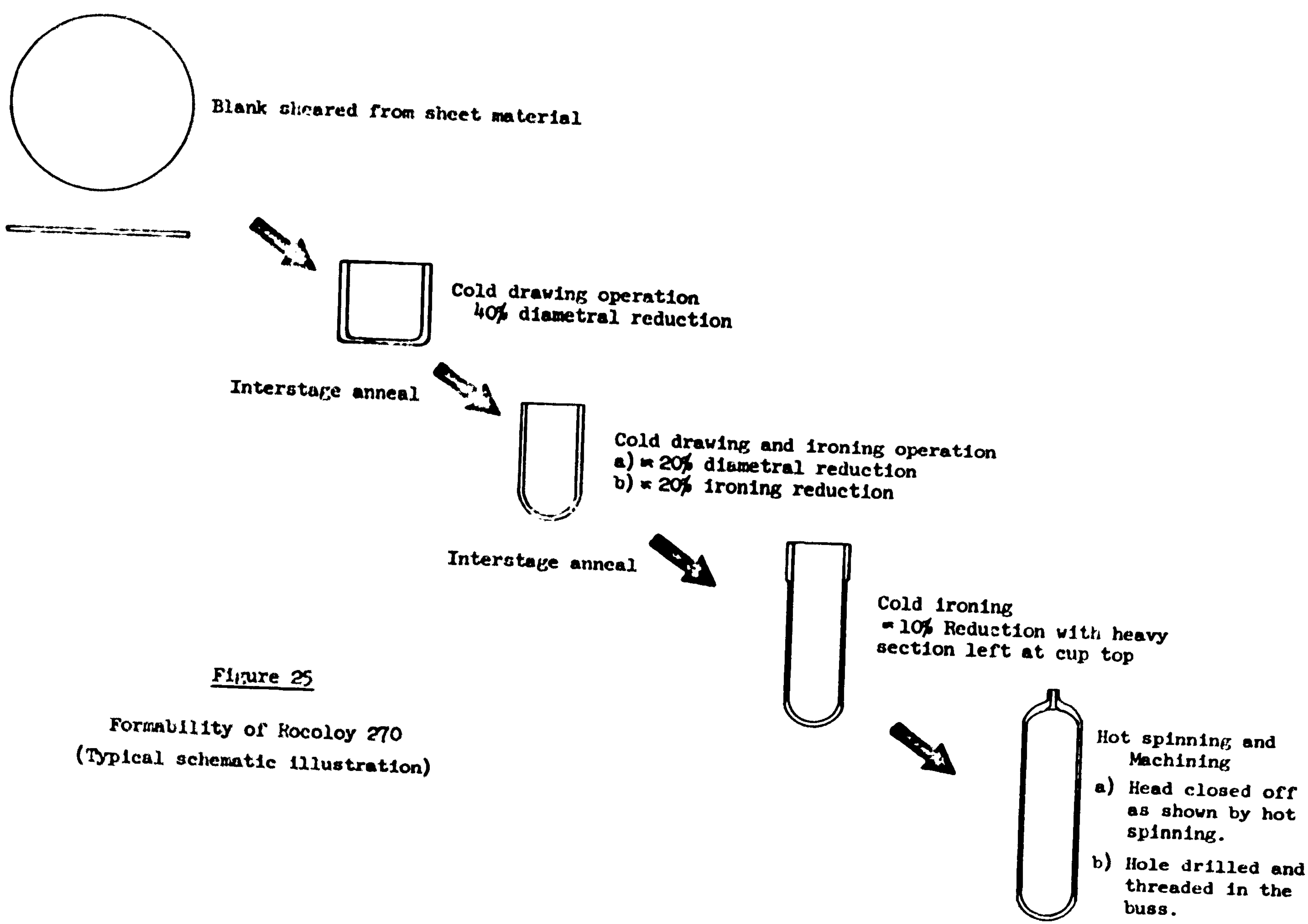


Figure 25

Formability of Rocoloy 270
(Typical schematic illustration)

TABLE VII

Weld Bend Ductility of Rocoloy 270 Using MX-2 and 17-22 AS Filler Wires

Steel Thickness	Current Variations in Amperes	Heat Input in Joules per inch[1]	Bend Angle			Welding Characteristics			Hardness[2] Rockwell C Weld Metal	Type and Location of Failures
			Transverse		Longitudinal	First Pass	Second Pass			
			Face	Root	Face	Weld Contour	Weld Contour	Weld Penetration		
MX-2 Wire										
.040	25	2500	180	180	180	Good-Narrow		Good-Narrow[3]	33-36	None
.060	40	4000	180	180	180	Good-Narrow		Good-Narrow	34-36	None
.100	65	11,700	180	180	180	Low	Good	Good	33-36	None
17-22 AS Wire										
.040	25	2500	180	180	180	Good-Narrow		Good-Narrow	35-37	None
.060	40	4000	180	180	180	Good-Narrow		Good-Narrow	33-38	None
.100	65	11,700	180	180	180	Concave	Fair	Good	33-36	None

[1] All three welded at 15 volts, .040 and .060 at 9 inches per minute and .100 at 5 inches per minute. Preheated to 400 F.

[2] Hardnesses taken on welds after stress relieving at 1325 F for 1/2 hour.

[3] .040 and .060 in. thick, one pass weld only.

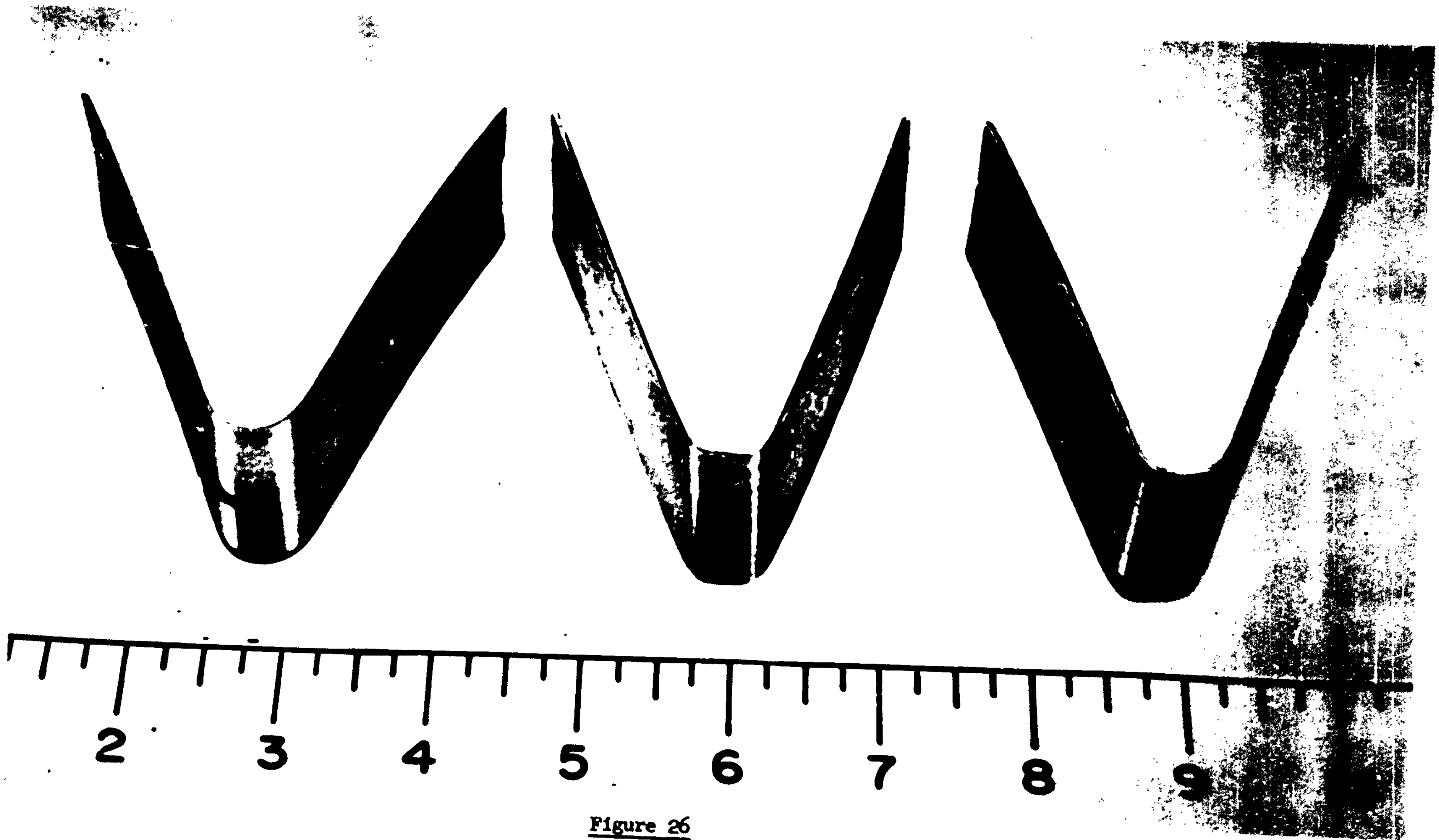


Figure 26

Bend Ductility of Stress Relieved Butt Welds on Rocoloy 270
Longitudinal Face Bend, Transverse Face Bend, Transverse Root Bend
Sheet Thickness 0.040 in.

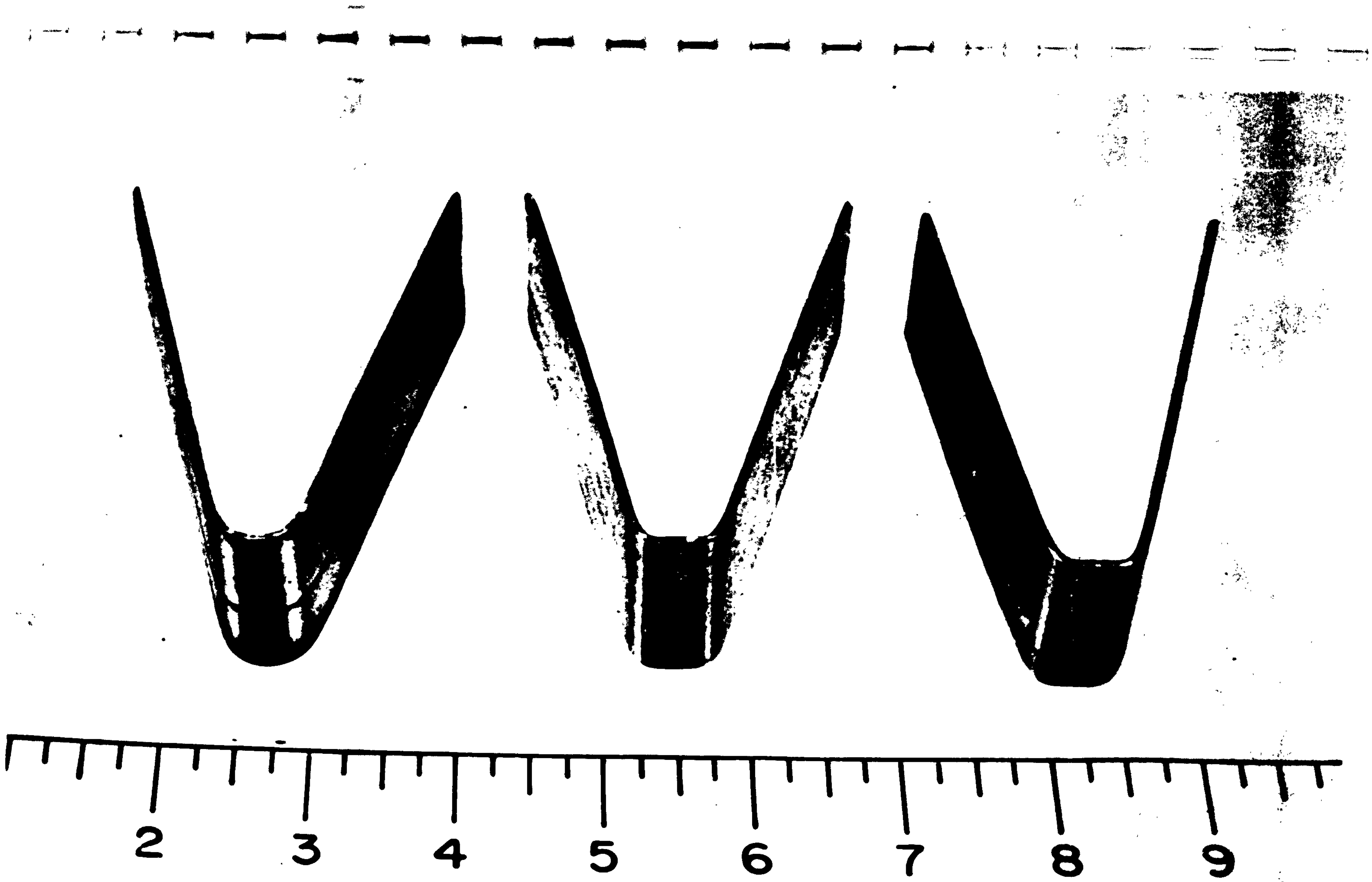


Figure 27

Bend Ductility of Stress Relieved Butt Welds on Rocoloy 270
Longitudinal Face Bend, Transverse Face Bend, Transverse Root Bend
Sheet Thickness 0.060 in.

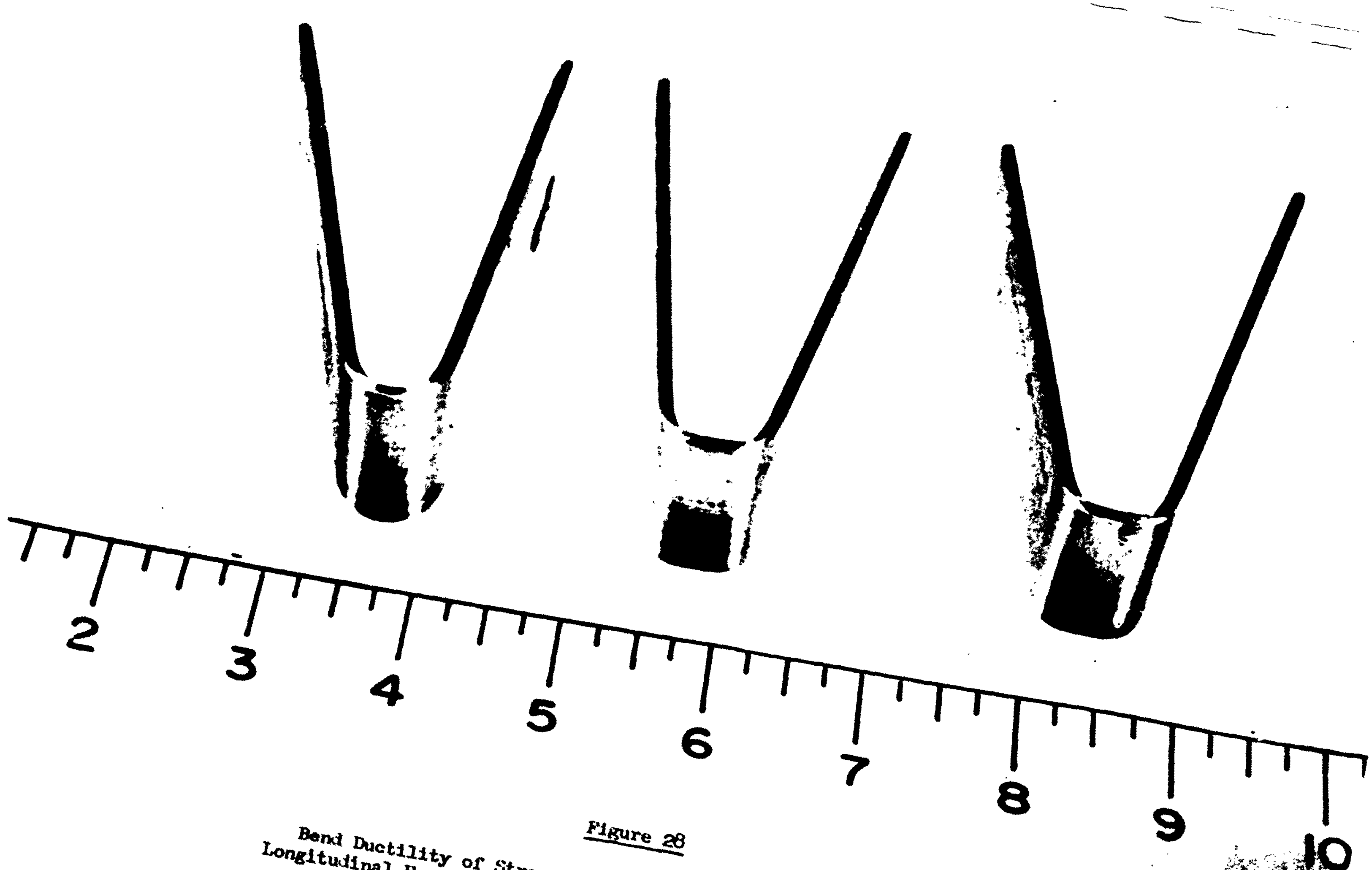


Figure 28

Bend Ductility of Stress Relieved Butt Welds on Rocoloy 270
Longitudinal Face Bend, Transverse Face Bend, Transverse Root Bend
Sheet Thickness 0.100 in.

TABLE VIII

Tensile Properties of Longitudinal and Transverse Butt-Welded Tension Coupons of Rocoloy 270 Using MX-2 and 17-22 AS Filler Wires

Material Thickness	Trans. or Long. Welds	Filler Wire	0.2% offset Yield Strength ksi [1]	Tensile Strength ksi	Elongation Per Cent in 1 in. [2]
.100	T	MX-2	309	322	5.0
	T		277	310	5.0
	T		269	313	6.0
	L		268	316	8.0
	L		272	317	3.0
.060	T	MX-2	271	317	
	T		290	334	
	T		267	305	2.0
	L		265	307	5.0
.040	T	MX-2	255	294	3.0
	T		315	358	2.0
	T		258	266	
	L		247	282	3.0
	L		259	299	4.0
.100	T	17-22 AS	286	315	6.0
	T		285	308	2.0
	T		281	325	7.0
	L		263	290	
	L			250	1.0
.060	T	17-22 AS	272	314	2.0
	T		282	315	3.0
	T		291	322	2.0
	L		266	314	7.0
	L		260	307	4.0
.040	T	17-22 AS	251	284	2.0
	T		251	295	3.0
	T		266	306	2.0
	L		249	292	
	L		269	311	5.0

[1] All specimens austenitized at 1730 F, double tempered at 600 F.

[2] No per cent Elongation indicates fracture outside gage length.

All welded tension test coupons of these studies failed in the parent material, about 1/8 in. to 1/2 in. away from the weld joint.

Macrographs of machine TIG butt welds made on 0.060 in. thick sheets of Rocoloy 270 using MX-2 filler wire are shown in Figure 29 for three heat treated conditions.

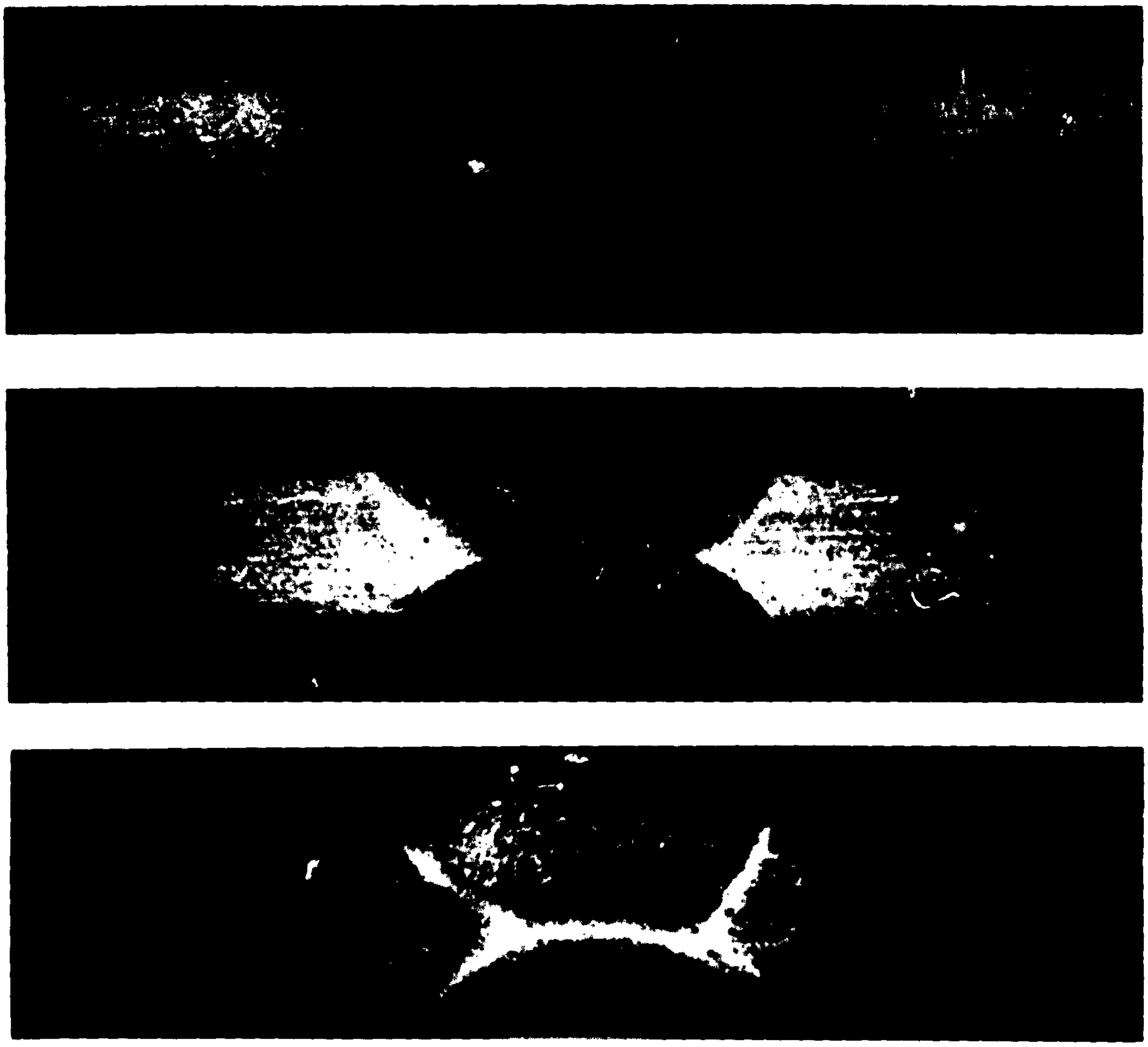
WELD GRAIN REFINEMENT STUDIES USING ULTRASONIC VIBRATION

Weld grain refinement studies using ultrasonic vibration were conducted in an attempt to improve efficiency and reliability of welded seams in ultrahigh-strength steel motor cases. In the early stages of POLARIS motor case development, welding of high-strength steels was considered to be a difficult fabrication procedure and a majority of motor case failures were attributed to lack of strength, ductility, and other defects in welded joints. Some of these failures invariably occurred in the weld "heat affected" zone. The poor efficiency of the weld joints was attributed to large grain size in the weld deposit.

These problems were later rectified by adopting multiple pass welding techniques which, however, resulted in increased fabrication costs. It was then thought that some method of weld grain refinement during solidification of welds may be of considerable value to this program. The feasibility of using ultrasonic vibration for this purpose was therefore investigated.

At first, studies were directed toward development of an ultrasonic generator and a transducer-coupler. These experiments were conducted at Aeroprojects, Inc., Westchester, Pa., as a subcontract.

The experimental setup used consisted of a welding fixture assembled with copper V-rollers which accommodated the ultrasonic transducer-coupler system riding on a tubular specimen as shown in Figure 30. The power source for the transducer was a 600 watt electronic generator.



(a)

(b)

(c)

Figure 29

Macrographs of Machine TIG Welds Made on Rocoloy 270, 0.060 in. thick Sheets using MX-2 Fillerwire (15X)

(a) As Welded, Stress Relieved 1325 F, 1/2 Hr.
(b) Weld as Quenched from 1730 F.
(c) Weld Quenched and Double Tempered at 600 F.

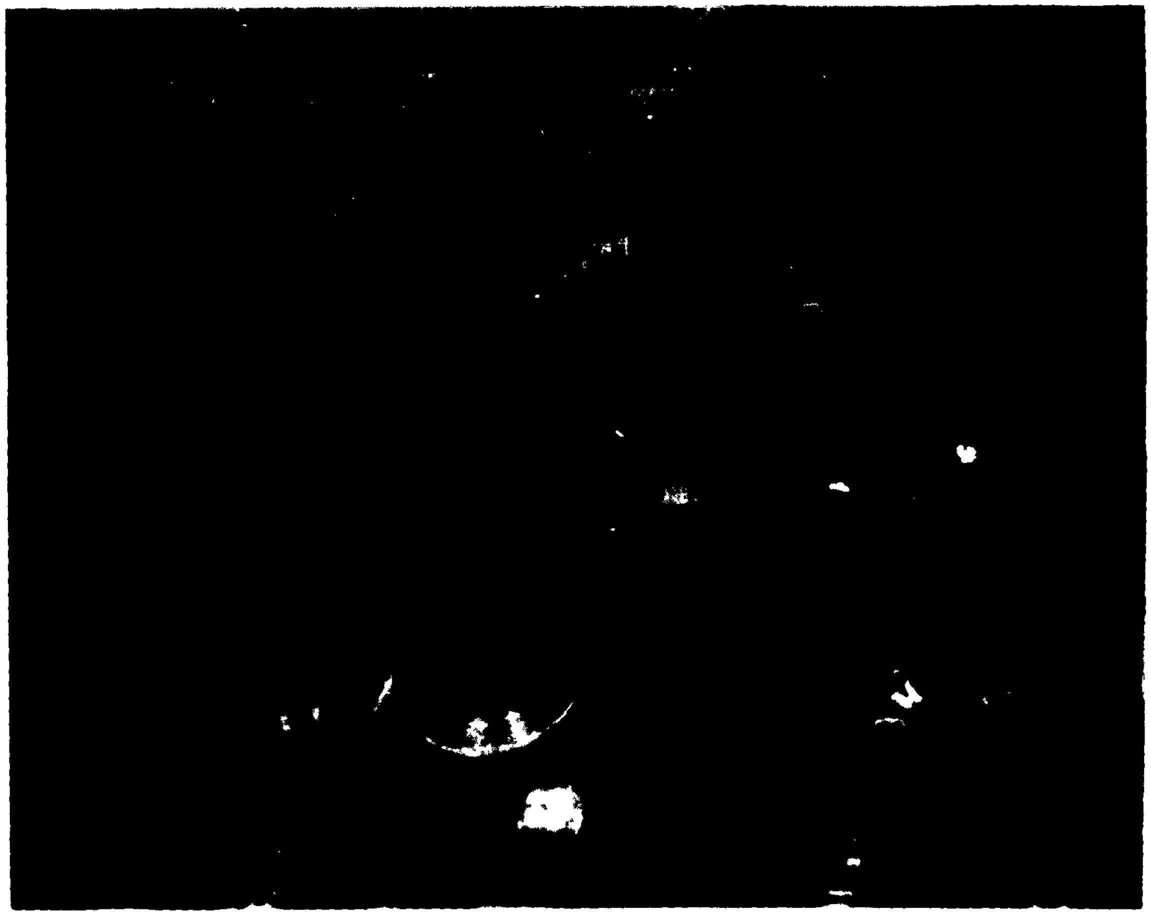


Figure 30

15 K_c Ultrasonic Vibration Set Up Used in Weld Grain Refinement Studies.

Test parameters investigated for various specimens are shown in Table IX. The results of metallographic examination of welds are summarized in Table X. These studies led to the conclusion that ultrasonic vibration of welds during solidification can achieve grain refinement in the weld bead. The ultrasonic power input and vibrational frequency required are 40 watts to 85 watts at 15 KC to 28 KC, respectively, for successful grain refinement of weld beads of thickness ranging from 0.050 in. to 0.150 in.

For evaluation of the response of this weld grain refinement technique in various steels and other nonferrous high-strength alloys, a 300 watt generator and 25 KC transducer-coupler were procured from Aeroprojects, Inc.

Initial experiments with this equipment were conducted on welds placed on flat plates simulating longitudinal seam welds in a rolled missile motor shell. Since the longitudinal seam welds became unpopular during the course of development of A 2 second stage POLARIS motor cases, later work on weld grain refinement was diverted to girth welds.

The experimental setup designed and built at Mellon Institute for this purpose is shown in Figure 31. A number of girth welds were made on cylindrical vessels and rings cut from deep drawn motor case shells varying in diameter from 6 in. to 14 1/2 in. Of the various test parameters, the generator output was varied while keeping constant the current, the welding speed, the speed of rotation of work piece and the ultrasonic wave frequency. The weld grain refinement achieved in MX-2 steel welds can be seen from macrostructures presented in Figure 32 through 36. Macrostructure shown in Figure 32 represents grain structure in a normal single pass TIG weld. Weld macrostructures shown in Figures 33 through 36 illustrate the consequence of application of ultrasonic energy in increasing order of magnitude. Grain refinement in the welds appears best at power input levels 120 watts to 240 watts.

The above experiments were then continued using a series of butted

TABLE IX

Test Conditions for the Various Tube Sections

Tube No.	Frequency Kilocycles	Tube Sections *A	B	C	D
T-1	15	100 watts	100 watts	100 watts	
T-2	15	Alternately 3" control and 3" 300 watts ultrasonic	150 watts		
T-3	15	75 watts			
**T-4	15	75 watts. No clamping force then 25 watts			
T-5	28	30 watts	50 watts	85 watts	
T-6	28	30 watts	50 watts	85 watts	
7	28	40 watts	40 watts		
8	28	40 watts	40 watts		
9	28	40 watts	3" at 40 watts 4" at 85 watts		
10	28	40 watts	85 watts	85 watts	1st part control then 40 watts
11	28	Control	40 watts	85 watts	1st part 40 watts then 85 watts
12	15	Control	40 watts	70 watts	
13	15	Control	Control	Control	
14	15	Control	40 watts	40 watts	40 watts to 70 watts changing gradually
15	15	40 watts	Control then 40 watts	1-7/8" 70 watts 2-1/2" pulsing 1-1/2" 70 watts 1-1/2" 40 watts 1-1/2" control	

* A, B, C, D, refer to 1 ft. sections of the tube weldment.
** Clamping force was 50 pounds throughout the experiment except for tube No. T-4.

TABLE X

Results of Metallographic Examination of Various Tube Sections

Specimen No.	Grain Size	Grain Shape	Imperfections	d(cm)	Remarks	Welding Conditions
T-2-A	Small-Med	Equiaxed and Columnar		1.05	Smooth bead, excess deposit	Non-Ultrasonic Control
T-2-B	Small	Equiaxed		1.30	Smooth bead, excess deposit; off-center arc	150 w, 15 kc
T-4-A	Med	Columnar and Equiaxed		1.05	Irregular top bead	25 w, 15 kc
T-4-B	Med	Columnar		0.85	Smooth	Non-Ultrasonic Control
T-6-A	Med-Large	Columnar and Equiaxed		0.90	Smooth	30 w, 28 kc
T-6-B	Small-Large	Equiaxed		1.00	Rough bead	50 w, 28 kc
T-6-C	Med	Columnar and Equiaxed		0.95	Smooth	85 w, 28 kc
7-B	Large	Columnar		0.90	Irregular top bead	40 w, 28 kc
7-B	Large	Columnar		1.10	Irregular top bead	40 w, 28 kc
11-A	Large	Columnar		1.20	Smooth bead	Non-Ultrasonic Control
11-B	Large	Columnar		0.90	Irregular top bead	40 w, 28 kc
11-B	Med	Equiaxed		1.10	Slightly irregular top bead	40 w, 28 kc
11-C	Med	Equiaxed		1.05	Slightly irregular top bead	85 w, 28 kc

Continued......

TABLE X (Continued)

Specimen No.	Grain Size	Grain Shape	Imperfections	d(cm)	Remarks	Welding Conditions
11-C	Small	Equiaxed		1.00	Slightly irregular top bead	85 w, 28 kc
13-A	Med	Partly Columnar		1.10	Smooth, regular bead	Non-Ultrasonic Control
14-A	Med	Columnar		0.95	Incomplete fusion	Non-Ultrasonic Control
14-B	Small	Equiaxed	Voids		Very irregular bead	40 w, 15 kc
14-C	Small	Equiaxed		1.00	Very rough bead	40 w, 15 kc
14-C	Med-Small	Equiaxed		0.95		40 w, 15 kc
15-B	Med	Partly Columnar			Smooth bead	Non-Ultrasonic Control
15-B	Med	Partly Columnar			Smooth bead	Non-Ultrasonic Control
15-C	Small	Equiaxed	Cracked	1.15	Rough bead	70 w, 15 kc pulsed
15-C	Small	Equiaxed	Cracked	1.15	Rough bead	70 w, 15 kc pulsed
15-C	Small	Equiaxed	Cracked	1.15	Very rough bead	70 w, 15 kc pulsed
15-C	Small	Equiaxed	Cracked	1.15	Very rough bead	70 w, 15 kc pulsed
15-C	Small	Equiaxed	Cracked	1.15	Irregular bead	70 w, 15 kc pulsed
15-C	Small	Equiaxed	Cracked	1.10	Irregular bead	70 w, 15 kc
15-C	Small	Equiaxed	Cracked	1.05	Very irregular bead	70 w, 15 kc
15-C	Small	Equiaxed	Cracked	1.05	Incomplete fusion	40 w, 15 kc
15-C	Med-Large	Equiaxed and Columnar	Cracked	1.05	Slightly rough top bead	40 w, 15 kc
15-C	Med-Large	Equiaxed and Columnar	Cracked	1.05	Uniform bead; differential cooling	40 w, 15 kc

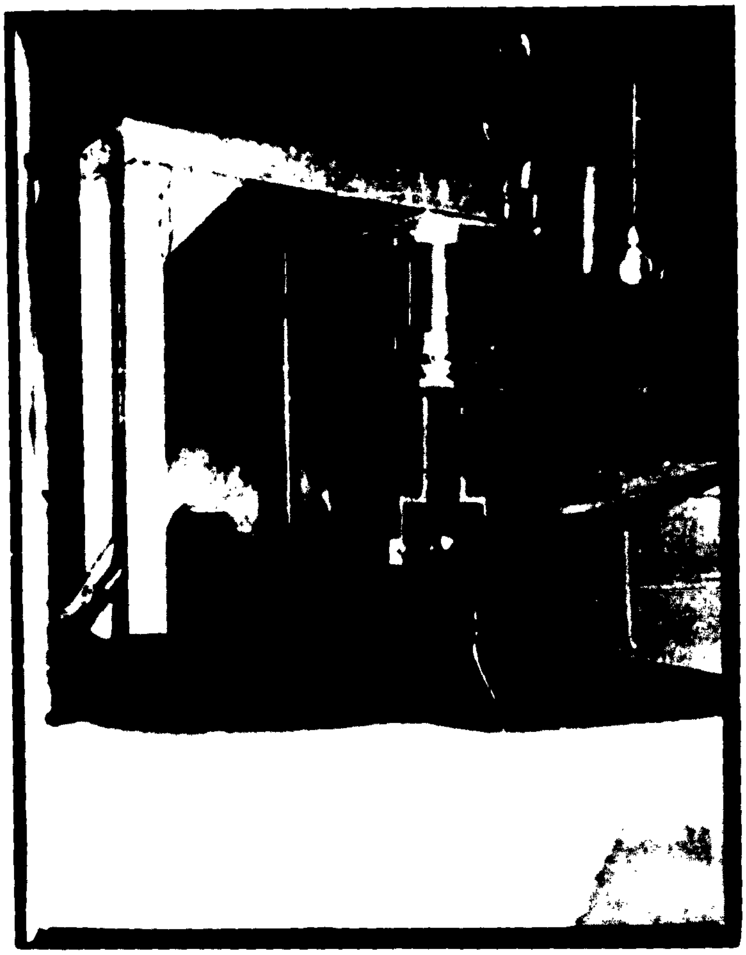


Figure 31

The Set-up Used in Experimentating with Ultrasonic Vibrations in Girth Welding.

Figure 32

A Weld in Which no Ultrasonic Vibrations were used.

Figure 33

Showing the Effect of the Ultrasonic Vibration on a Weld with the Generator Power Output set at 3.

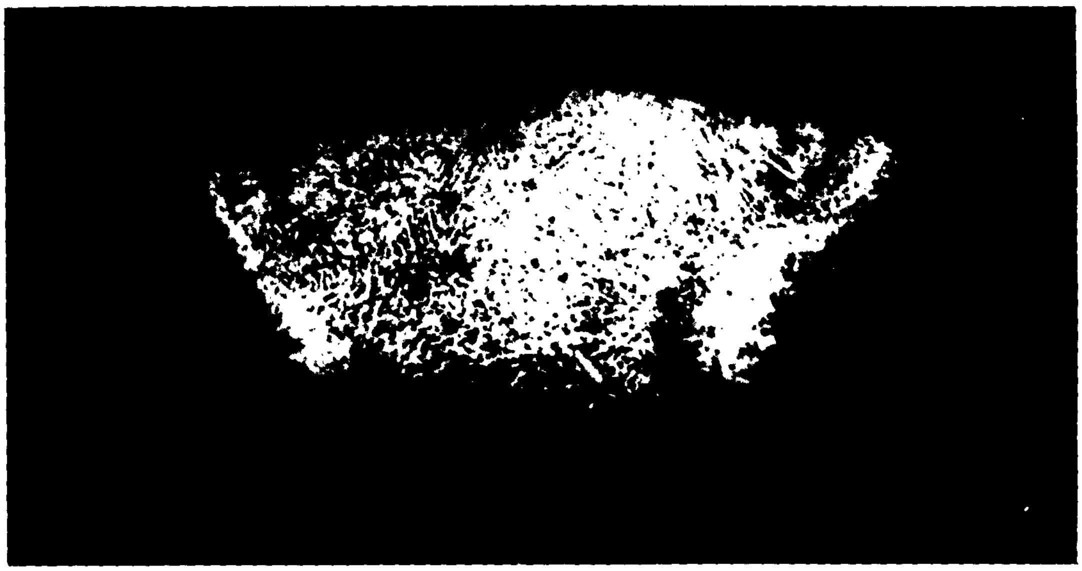


Figure 34

Showing the Effect of the Ultrasonic Vibration on a Weld with the Generator Power Output set at 5.

Figure 35

Showing the Effect of the Ultrasonic Vibration on a Weld with the Generator Power Output set at 6.

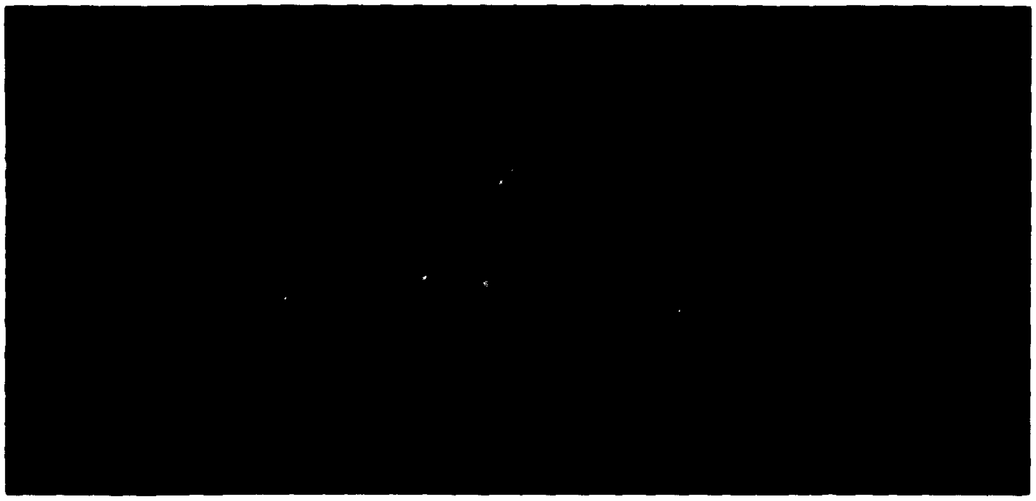


Figure 36

Showing the Effect of the Ultrasonic Vibration on a Weld with the Generator Power Output set at 7.

MX-2 steel rings and placing double pass TIG welds on the joints. One-half of each circumferential weld was made with the aid of ultrasonic vibration while the other half was welded without vibrating the weld bead. Also, in these experiments the transducer head was located at several positions with respect to the butt joint. The results of these studies have been shown macrographically in Figures 37 through 39. These pictures show the difference between cast structures for the two conditions. The grain refinement effected by the application of ultrasonic energy entails breaking up the columnarity in the solidified weld bead and the production of nearly uniform equiaxed crystals.

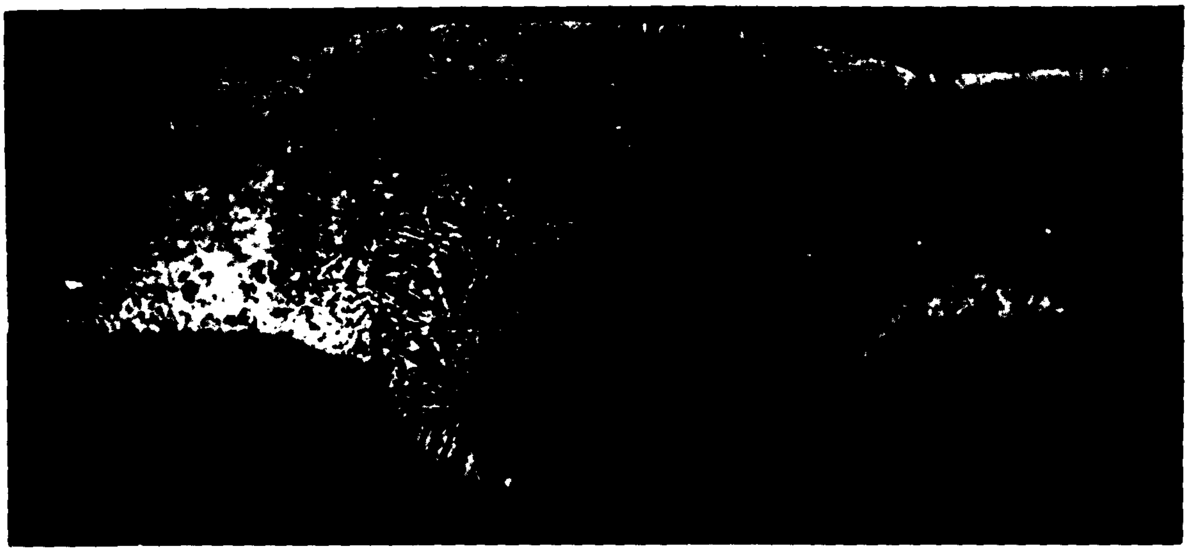
It was evident from these and subsequent experiments, that in girth walding, uniform energy transfer during ultrasonic excitation could be best achieved by positioning the probe on the weld puddle trailing the arc at a specified distance dictated by the freezing characteristics of the weld filler wire composition and bead thickness.

A few tensile tests conducted on welded specimens whose welds were refined with the aid of ultrasonic energy indicated one hundred per cent joint efficiency and strength in excess of that of the parent material. The fractures were well outside the heat affected zone. These tests, however, were insensitive for direct evaluation of the efficacy of this welding technique.

It was therefore decided to conduct fatigue tests on welds with and without grain refinement by ultrasonic means. Welded sheet fatigue test specimens were prepared and tested. The results obtained unquestionably showed the superiority of the ultrasonically treated weld joint.

ALUMINUM FINE WIRE DEVELOPMENT

In March, 1961, at the request of the contract monitor, new steel development phase of this program was redirected to include development of



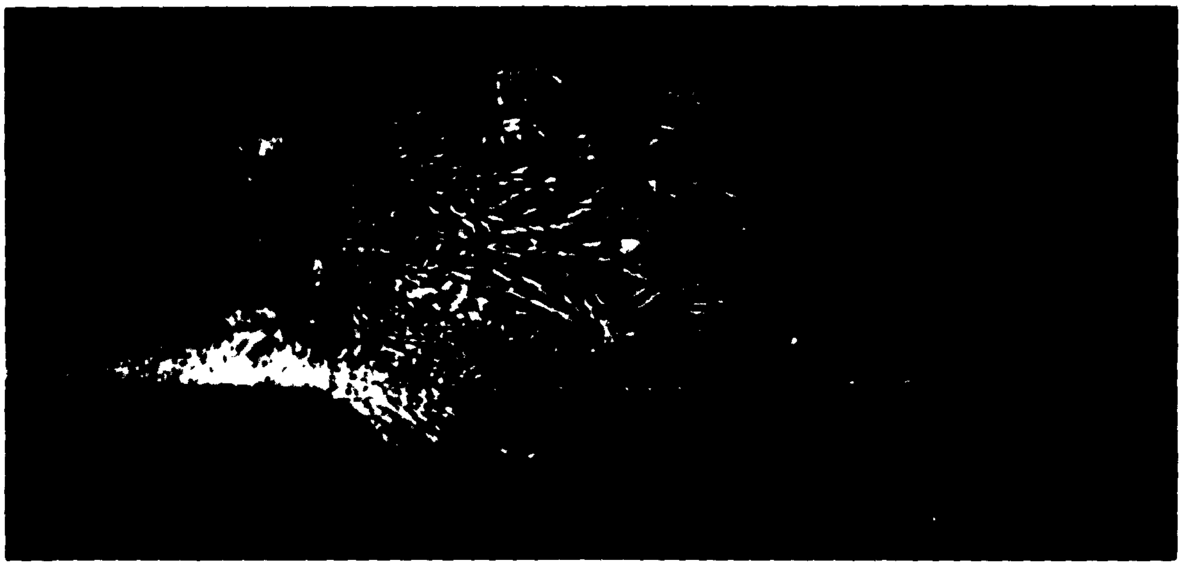


Figure 37

Photomacrograph of a girth weld. Top weld was ultrasonically agitated. Bottom weld with no ultrasonics applied.

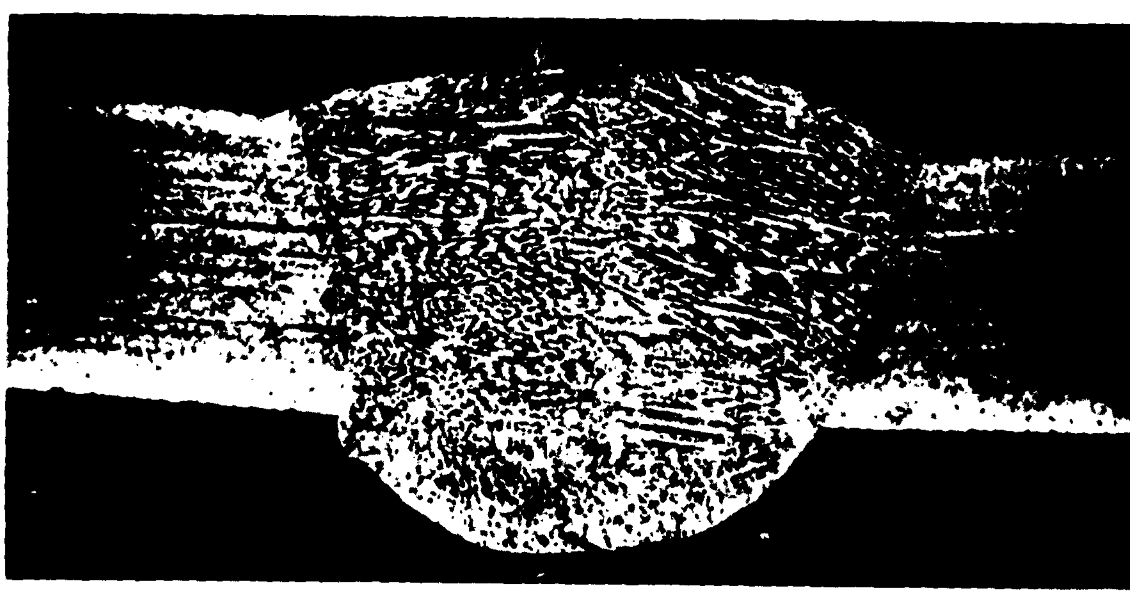


Figure 38

Photomacrograph of a girth weld. Top weld was ultrasonically agitated 2/8" from the edge of the weld. Bottom weld shows the same weld with no ultrasonics applied.

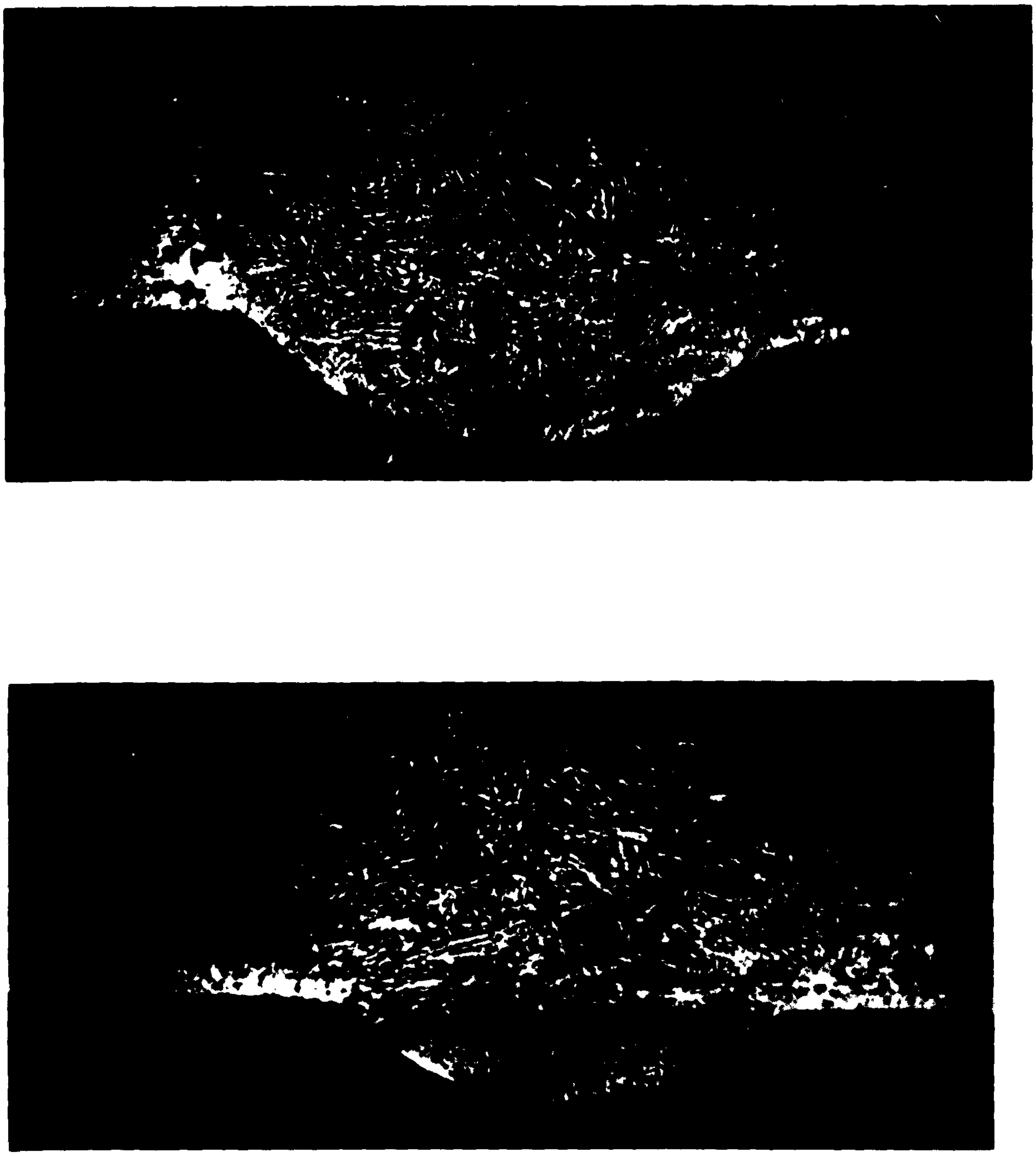


Figure 39

Photomacrograph of a girth weld. Top weld was ultrasonically agitated 5/8" from the edge of the weld. Bottom weld shows the same weld with no ultrasonics applied.

high-strength aluminum alloy filaments of 0.002 in. to 0.008 in. diameter.

Accordingly, a program was immediately initiated to produce 0.005 in. diameter wire from aluminum alloys 2024-T4, 6061-T913 and 7075-T6. The final drawing of these alloy wires in continuous spooled length ranging from 5000 ft. to 12,000 ft. was successfully completed in May, 1961. Of these alloys, 2024 and 7075 require heat treatment in order to develop full strength and 6061 ages at room temperature after cold work to its full strength. Tensile test data obtained on these wires in the "as drawn" condition are shown in Table XI. It was then realised that these strength levels are significantly lower than expected and those needed for making a high-strength metallic wire wound epoxy bonded second stage POLARIS chambers.

One question which remained to be answered was the compatibility of metallic filaments with epoxy resins used in bonding fiberglass filament cases. It was decided to construct a sub-scale configuration of a missile case out of 6061-T-913 aluminum filament for determining its biaxial stress capabilities. Tooling for this was immediately available for constructing 5 1/2 in. diameter spheres and two such spheres as shown in Figure 40 were successfully built. Pressure testing of these spheres could not be done prior to the contract deadline date.

BERYLLIUM FINE WIRE DEVELOPMENT

Since the aluminum wires showed little promise as to development of high strength, the contract monitor during May, 1961, requested initiation of work towards development of beryllium fine wires. This program was readily accomplished from the experience gained in the aluminum wire drawing program. However, beryllium wire in continuous spooled length in excess of 2000 ft. could not be drawn. It was realized that a research program on methods of

TABLE XI

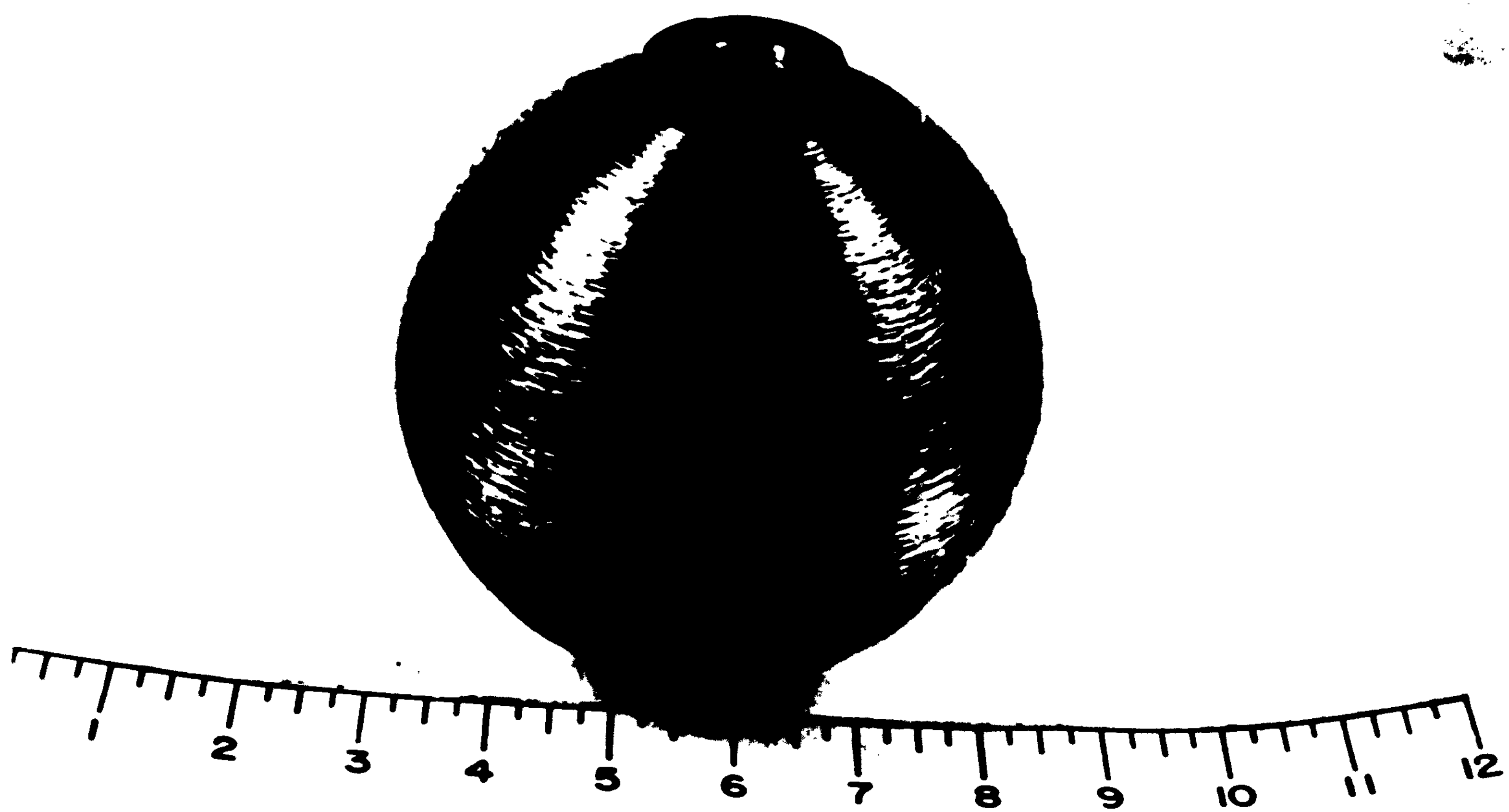
Room Temperature Tensile Properties of Aluminum Wires

(0.005 in. dia.)

Alloy Designation	Specimen Number	0.2% offset Yield Strength ksi	Ultimate Tensile Strength ksi
7075[a]	1	54	62
	2	56	62
	3	56	61
6061-T-913[b]	1	73	76
	2	74	76
	3	73	76
2024[a]	1	54	56
	2	54	59
	3	54	58
	4	54	59

(a) As drawn un-heat treated condition.

(b) Aged at room temperature to full strength.



<u>Figure 40</u>

Aluminum Filament Wound Sphere.

avoiding undesirable grain orientation and development of suitable heat treatment procedures is necessary prior to successfully drawing beryllium wire in lengths exceeding approximately 2000 ft. Tensile properties of the beryllium wire were next determined and the data are given in Table XII.

With the completion of work on beryllium wire, this program terminated on September 30, 1961.

Respectfully submitted,

GKBhat

G. K. Bhat
Project Leader

Approved:

H L Anthony

H. L. Anthony III
Director of Research

TABLE XII

Room Temperature Tensile Properties[a] of Beryllium Wire[b]

(0.004 in. Dia.)

Test Number	Threshold[c] Yield Strengh ksi	0.2% offset Yield Strength ksi	Ultimate Tensile Strength ksi
1	144	166	177
2	136	166	179
3	144	168	178

(a) Tests conducted in accordance with ASTM specifications.

(b) As-drawn condition with coating on surface.

(c) Corresponds to 0.05% offset.

APPENDICES

APPENDIX A

Experimental Steels[1]

Chemical Composition[2], Per Cent

Designation	C	Mn	Si	Cr	Ni	Mo	W	V	Co	Others
MX-16	0.44	0.54	1.51	1.20	--	0.30	--	0.14	1.34	--
MX-17	0.44	0.74	1.24	1.21	--	--	0.68	0.13	0.95	--
MX-18	0.44	1.00	1.20	--	--	--	0.70	0.16	--	--
MX-19	0.47	0.70	1.21	1.38	--	--	0.66	0.22	1.45	--
MX-20	0.41	0.84	0.80	--	0.04	--	--	0.14	1.59	--
MX-21	0.58	0.68	1.21	1.34	--	0.45	--	0.19	2.44	--
MX-22	0.47	1.20	1.23	1.46	--	--	0.62	0.19	1.02	--
MX-24	0.39	1.41	0.74	--	0.05	--	--	0.14	1.52	--
MX-25	0.39	1.41	0.77	--	--	--	--	0.11	2.59	--
MX-26	0.41	0.84	0.75	--	1.70	--	--	0.17	1.52	--
MX-27	0.41	0.79	0.99	1.16	--	0.59	--	0.15	1.19	--
MX-28	0.39	0.84	0.80	0.82	0.56	--	--	0.14	1.12	--
MX-29	0.39	0.81	0.77	0.87	1.13	0.16	--	0.13	0.13	--
MX-30	0.42	1.15	0.92	0.88	1.93	0.32	--	0.06	2.02	--
MX-31	0.52	0.55	1.02	1.17	0.40	0.38	--	0.13	2.84	--
MX-32	0.42	0.64	1.03	3.14	--	1.35	--	0.40	0.94	--
MX-34	0.37	0.67	0.31	1.09	--	0.40	--	0.19	1.20	0.26 Be
MX-35	0.37	0.67	0.41	1.95	--	0.68	--	0.20	2.80	0.68 Be
MX-41	0.42	0.70	1.20	1.50	0.50	0.20	0.60	0.20	1.50	--
MX-42	0.41	0.70	1.20	2.00	1.00	0.30	0.60	0.40	2.00	--
MX-43	0.38	0.50	0.90	3.00	--	0.40	1.00	0.30	2.00	--
MX-44	0.41	0.60	1.20	1.30	0.20	--	0.70	0.18	1.35	--
MX-45	0.40	0.60	1.00	1.50	0.40	0.50	1.00	0.60	1.00	0.30 Cb
MX-46	0.40	1.50	0.80	1.50	0.30	0.60	0.60	0.30	1.00	0.50 Ti
MX-51	0.50	0.40	1.25	1.25	1.25	0.75	--	0.12	2.00	--
MX-52	0.55	0.40	1.25	1.10	--	1.00	--	0.15	2.25	--
MX-53	0.62	0.30	1.50	1.00	1.50	0.50	--	0.07	3.00	--
MX-54	0.55	0.40	1.25	1.60	3.00	0.60	--	0.06	4.00	--
MX-55	0.45	0.60	1.20	1.25	0.40	0.30	0.40	0.15	1.50	--
MX-56	0.45	0.50	1.00	1.10	2.20	0.60	1.20	0.15	--	--
MX-57	0.45	1.20	1.00	1.00	1.35	--	0.50	0.12	1.40	--
MX-58	0.48	0.90	1.45	1.00	1.20	0.50	--	0.20	1.00	--
MX-61	0.46	1.60	1.20	1.15	0.50	0.50	--	0.30	1.60	--

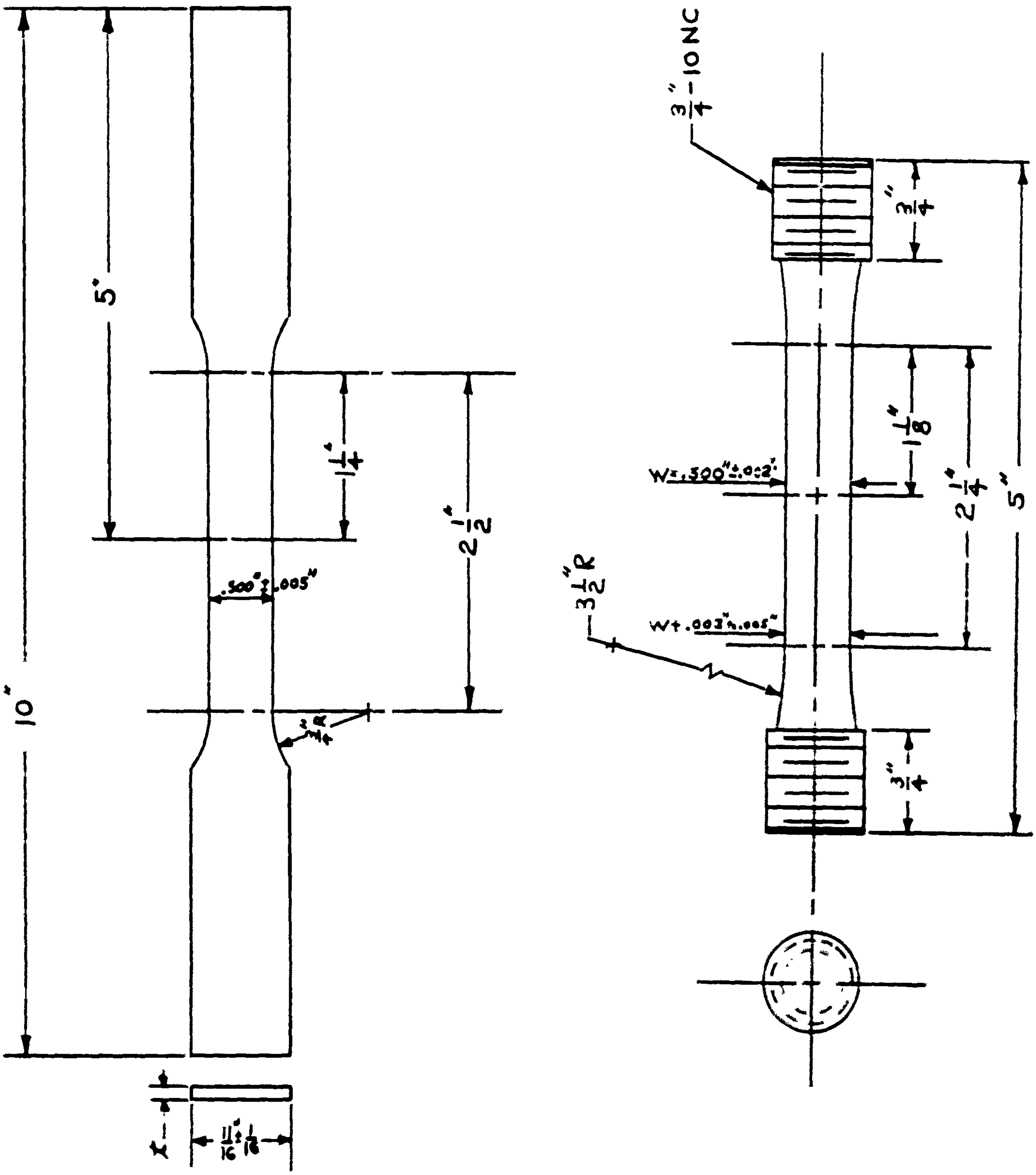
[1] Size of Heats

MX-19 through MX-32 - 400 lb. air induction furnace heats.
MX-34 and MX-35 - 600 lb. air induction furnace heats.
MX-51 through MX-54 - 1000 lb. vacuum induction furnace heats.
MX-16, MX-17, MX-18, MX-41 through MX-46, MX-55 through MX-61 - 2000 lb. electric arc furnace heats.

[2] Sulfur and phosphorus in all the above heats were each less than 0.013 per cent.

APPENDIX I

Tension Test Specimens - Sheet and Bar



APPENDIX II

Edge Notch Tensile Test Specimens